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1. TOPIC 1

Paragraph 1.

Paragraph 2.

1.1 Sub Topic 1

Paragraph 1.

Paragraph 2.

2. TOPIC 2

Paragraph 1.

Paragraph 2.



Figure 1: Title of figure.

Table 1: Title of table.

Content	Content	Content
Content	Content	Content
Content	Content	Content
Content	Content	Content

Equation 1 (1)
Equation 2 (2)

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Online Sources

GTOPO30 (1996). *GTOPO30: Global 30 Arc Second Elevation Data Set*. Available online at: <http://edcwww.cr.usgs.gov/landdaac/gtopo30/gtopo30.html> (Last access date: 1 June 2009).

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Wood, J. (1996). *The Geomorphological Characterization of Digital Elevation Models*. PhD Thesis, Department of Geography, University of Leicester, Leicester.

EFFECT OF STITCHING PARAMETERS ON BALLISTIC RESISTANCE OF KEVLAR FABRICS

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ABSTRACT

There may be advantages and disadvantages of stitching in soft body armours. The stitching process is often a time consuming and costly step in manufacturing. It is assumed by most armour designers that stitching can create weak points (at the stitched points), and also reduce armour flexibility to users, resulting in comfort issues. This study investigates the relationship between soft body armour stitching parameters with ballistic resistance test results (blunt trauma depth) using the Design of Experiments (DOE) approach. A Response Surface Methodology Design was carried out to identify the factors that influence the ballistic impact resistance of stitched fabric armour. The factors that were studied were stitching perimeter, number of fabric layers and thread strength. The ballistic test was conducted with reference to the NIJ 0101.08 standard. The developed statistical model showed that the number of fabric layers and stitching perimeter have significant influence on ballistic resistance capability as compared to the others factors. This study can be a reference for body armour manufacturers for considering the level of stitching to be applied during the soft body armour production process.

Keywords: *Fabric armour; stitched fabric; blunt trauma depth; design of experiment (DOE); Response Surface Methodology Design.*

1 INTRODUCTION

Researchers are always looking for new materials to improve the functional performance of soft body armour. Body armour has been in use for all of recorded history, beginning with hides, leather and bones, progressing to bronze, steel, ballistic cloth, ceramics and lightweight composite. The modern bullet proof vest has primarily been a way to protect oneself from projectile threats in domestic crime and military engagements. The first type of non-glass type of ballistic resistance fibre was KEVLAR, which was made from aramid constituents by Du Pont in the 1960s. KEVLAR has a long chain like molecule known as polymer that consists of repeating unit cells called monomers. KEVLAR fibre is an array of molecules oriented in parallel to each other (Clements, 1998). Typically, the standard military personal armour system includes aramid based fabric material, with or without ceramic inserts (Kobi *et al*, 2006; Karahan *et al.*, 2008; Abdulghaffar, 2015), where the ballistic resistance capability of the armour is measured by measuring the blunt trauma depth. Blunt trauma is a prominent assessment criteria for body armour standard because when the armour is penetrated and is pushed into the wearer's body, the body wall is pressed inward instantaneously with great tenacity (Hanlon & Gillich, 2012).

The contributing factors in the performance of soft body armour in order to be able to defeat projectiles can be in the form of fibres and yarn properties, fabric construction, fabric weight per unit area, and ply number. These parameters were actively studied by Chocron-Benloulo *et al*, (1997), Lim

et al., (2002) and Ahmad *et al.* (2008). From these studies, it can be noted that evidences exist which report that stitching in a soft body armours panel impairs the ballistic resistance properties of the panel (NIJ Standard-0108.01,1985). Stitching may decrease in-plane stiffness and tensile properties by varying amounts. The thread and needle used for stitching can damage the microstructure by breaking, spreading and crimping the fibres around the stitch holes. The performance of fabric armours in ballistic impacts was reported to increase if the friction between yarns was increased. Thus, the use of stitching was assumed to contribute to the performance of body armours by reducing the inter-yarn friction in fabrics (Chocron-Benloulou *et al.*,1997; Abdulghaffar, 2015).

From the literature study conducted, there are studies (Ahmad *et al.*, 2008; Karahan *et al.*, 2008; Wang *et al.*, 2014; Rana *et al.*, 2014; Xiaogang *et al.*, 2014; Dariush *et al.*, 2016) that have been conducted to examine the behaviour of stitched fabrics, but only a few models have been developed to quantify the effect of stitching in terms of body armour performance. Yahaya *et al.* (2010) developed a soft body armour energy absorbing model based on fabric areal density (represents the number of plies of fabric), stitched area, stitch density, sewing thread strength and bullet velocity. The model showed that fabric areal density, stitch density and bullet velocity have the most influence on the stitched fabric's energy absorption capabilities. Body armour manufacturers tend to use similar thread materials as the ballistic resistance fabric's material in their production. Thus, the stitching effect towards the quality of soft body armour is of great interest to be studied. Furthermore, there is a need for a comprehensive experimental study to determine the effect of the stitching parameters that contribute to the performance of stitched fabrics in body armours. This study attempts to investigate the soft body armour stitching parameters (stitching perimeter, numbers of fabric layers and thread strength) that influences the blunt trauma depth.

2. MATERIAL AND METHODS

For this study, a Kevlar Type 802F sheet was cut into 10 x 10 in pieces and the multi-layer fabric was stitched together for the ballistic test using an industrial sewing machine (as shown in Figure 1). The stitching patterns used in this study are commonly used in the local soft body armour manufacturing industry. The detailed material specification is shown in Table 1. A lock stitch pattern was used to stitch multiple plies of fabric into the test samples. The yield strength of several thread materials was determined in accordance to ASTM D2256 (Test Method for Tensile Properties of Yarns by the Single-Strand Method) using a Tinius Olsen model HKS50 universal testing machine. The tensile testing was performed using strain rate of 0.1 mm/min with a load cell of 500N capacity at room temperature of 20 °C and relative humidity of 65%.

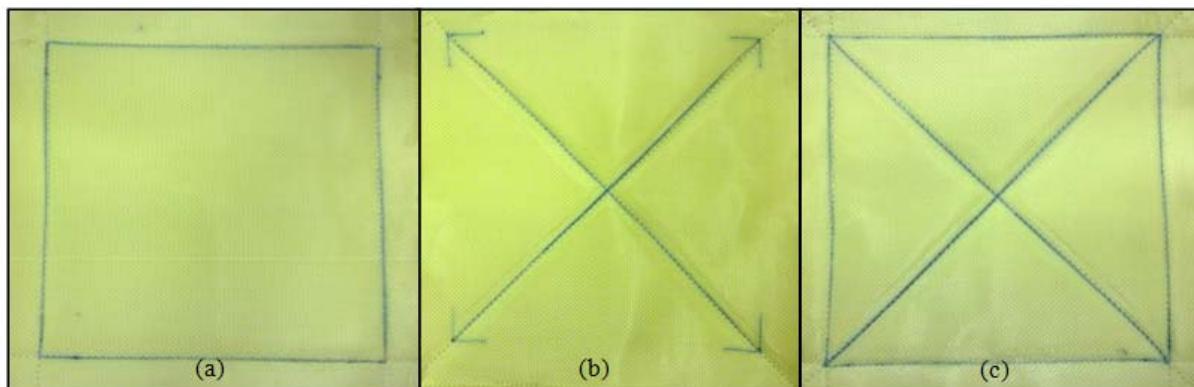


Figure 1: Stitched fabric panels for the ballistic test with various designs: (a) perimeter, (b) cross and (c) cross and perimeter.

Table 1: Product specification of the Kevlar fabric used in this study.

Description	Specification
Style	802F
Decitex	1100
Weave	Plain
Thread: Warp/Weft (E/10cm)	85
Weight (g/m ²)	185

A standardised ballistic testing method with reference to the United States NIJ Standard 0101.08 (1985) was used to determine the ballistic resistance capability of the protective materials. The testing was conducted at a 600 m indoor ballistic test range using a STZA 12A test gun at the Science and Technology Research Institute for Defence (STRIDE), Batu Arang, Selangor, as shown in Figure 2. The ammunition used in the test procedure was 9 mm Full Metal Jacket (FMJ) as specified in the above mentioned standard. The bullets used in the shootings had 8 g core weight and 9 mm diameter, with the impacting velocity adjusted to meet the range of ± 425 m/s. An array of chronographs were placed along the firing line in order to measure the initial projectile velocity. The stitched sample was attached to a backing material namely Roma Plastilina Type B (clay material) in order to measure the response in terms of blunt trauma depth, where the test samples were strapped as target. Trauma depth is defined as the allowable blunt trauma that a person can sustain (non-fatal) upon bullet impact while wearing body armour materials. According to NIJ Standard 0101.08, the allowable blunt trauma depth is at maximum of 44 mm.



Figure 2: The ballistic test setup (a) 9mm FMJ ammunition, (b) test gun, and (c) backing material and test sample holder.

By using the Response Surface Methodology Design, there were three main parameters that were investigated, which were thread strength, pattern parameter and fabric ply numbers. The fabric plies used for this study were 10, 15 and 20 plies. The experimental design for the study was formulated according to the selected factors and response as shown in Table 2.

Table 2: The Response Surface Methodology Design used for this study.

Factors	Coded	Low (-1)	Medium (0)	High (+1)	Response
Stitching Pattern Perimeter (mm)	A	Perimeter Stitched (0.575)	Cross Stitched (0.813)	Perimeter + Cross Stitched (1.388)	Blunt Trauma Depth
Thread Strength (MPa)	B	Jeans (9.67)	Polyester (32.14)	Nylon (134.62)	
Fabric Plies	C	10	15	20	

3. RESULTS & DISCUSSION

The blunt trauma depth was determined using a handheld laser measurement tool with ± 0.02 mm precision (as shown in Figure 3a). Based on the observations, if partial penetration (PP) occurred on the impacted point at the test sample, a distinct crater was formed in the backing material with irregular diameter and certain depth. The depth of this crater showed the tenacity of the bullet and the impact force transmitted to the back side of a panel. For complete penetration or perforation (CP), where the bullet travelled through the test sample, it was found that a hole with symmetrical diameter was formed at the backing material surface (as shown in Figure 3b).

The effect of stitching parameters subjected to projectile impact at a range from 380 to 399 m/s is as shown in Table 3. The 9 mm FMJ projectiles measurements were found to be inconsistent due to ammunition propellant imperfection, such as propellant material aging and decomposition, and different manufacturing batch number. This is generally common in any ammunition where the propulsion system is based from the instantaneous gas expansion in the cartridge due to combustion reaction of the propellant. However, the ballistic test was conducted in accordance to NIJ 0101.08 (Level II condition), in which projectile velocity can vary from 378 to 398 m/s. From Table 3, it can be also observed that Nylon thread exhibited greater blunt trauma depth reduction as compared to Polyester, followed by Jeans thread. This can be because the tensile yield strength of Nylon is superior to Polyester and Jeans threads.



Figure 3: Blunt trauma depth assessment: (a) Measurement of craters. (b) Comparison between complete penetration and partial penetration craters.

It is also observed that the stitching patterns also showed certain influence towards the blunt trauma depth reduction (ballistic resistance capability) of the test samples. The combination of perimeter and cross stitching pattern exhibit less blunt trauma depth readings as compared to perimeter, followed by cross stitched pattern. This can only imply that both stitch combinations provide more coverage area in term of inter-plies binding and therefore, allowing the test sample to resist the impact force uniformly. Furthermore, the combination of cross and perimeter stitching reduces the flexibility of the test sample (making it more rigid a compared to other patterns) and provides better ballistic resistance in terms of localised impact.

Table 3: Ballistic test results.

Thread Type	Number of Plies	Pattern Type	Impacting Velocity (m/s) (Mean \pm SD)	Result (CP/PP)	Blunt Trauma Depth (mm) (Mean \pm SD)
Polyester	10	Cross	395.5 \pm 10.6	CP	-
	10	Perimeter	381.0 \pm 2.8	CP	-
	10	Cross+Perimeter	397.0 \pm 2.8	CP	-
	15	Cross	388.5 \pm 0.7	PP	36.0 \pm 1.4
	15	Perimeter	385.5 \pm 2.1	PP	40.0 \pm 1.4
	15	Cross+Perimeter	392.0 \pm 2.8	PP	36.0 \pm 2.8
	20	Cross	393.0 \pm 0.0	PP	31.5 \pm 0.7
	20	Perimeter	388.0 \pm 8.5	PP	34.0 \pm 1.4
	20	Cross+Perimeter	391.5 \pm 2.1	PP	32.5 \pm 0.7
Nylon	10	Cross	390.0 \pm 11.3	CP	-
	10	Perimeter	395.0 \pm 2.8	CP	-
	10	Cross+Perimeter	396.0 \pm 11.3	CP	-
	15	Cross	394.0 \pm 2.8	PP	38.0 \pm 1.4
	15	Perimeter	389.5 \pm 4.9	PP	35.5 \pm 2.1
	15	Cross+Perimeter	394.5 \pm 6.4	PP	33.0 \pm 0.0
	20	Cross	388.0 \pm 2.8	PP	27.5 \pm 2.1
	20	Perimeter	398.0 \pm 2.8	PP	27.0 \pm 2.8
	20	Cross+Perimeter	395.5 \pm 3.5	PP	26.0 \pm 1.4
Jeans	10	Cross	396.0 \pm 4.2	CP	-
	10	Perimeter	399.0 \pm 1.4	CP	-
	10	Cross+Perimeter	399.5 \pm 10.6	CP	-
	15	Cross	395.5 \pm 12.0	PP	38.0 \pm 2.8
	15	Perimeter	393.5 \pm 10.6	PP	38.0 \pm 2.8
	15	Cross+Perimeter	388.5 \pm 4.9	PP	35.5 \pm 3.5
	20	Cross	398.5 \pm 3.5	PP	35.5 \pm 3.5
	20	Perimeter	398.5 \pm 3.5	PP	34.5 \pm 2.1
	20	Cross+Perimeter	398.0 \pm 0.0	PP	30.0 \pm 0.0

The obtained blunt trauma depth data from the ballistic tests were computed into a statistical model using the Design Expert 8 software. From the ANOVA results shown in Table 4, it can be observed that fabric plies (Factor A) are the main contributing factor for blunt trauma depth reduction followed by stitching pattern (Factor B) and thread strength (Factor C). This is because the ballistic resistance fabrics react as a defensive net like mechanism to stop the penetration tenacity of the projectile whilst the stitched area provides uniformity between the fabric plies. The relationship between the number of plies and stitching (Factor AB) also showed greater influence towards reducing blunt trauma depth as compared to other combinations. These analyses are in agreement with the physical observations (test sample damage mode and backing material indentation results). The results also concurred with the findings from other research works (Yahaya *et al.*, 2010; Othman & Hassan, 2013). Othman & Hassan (2013) reported that woven fabrics strength was observed to increase proportionally with increasing number of layers, whereas Yahaya *et al.* (2010) reported that areal density and stitching density contributed significantly towards the ballistic performance of Kevlar stitched fabrics.

From Table 4, the fit summary denotes that the quadratic model is more significant for the A-Layer parameter. The ANOVA model results show that the data is well fitted in this model blunt trauma depth optimization. The values of R^2 and Adj R^2 obtained are more than 85%. Therefore, it can be concluded that the regression model has produced a good indication on the relationship between the selected parameters to the blunt trauma depth at 95% confidence level. The predicted R^2 value is 71%, which means that the predicted model is slightly lower in accuracy as compared to the real results.

Table 4: ANOVA results of the tested samples.

Source	Sum of Squares	df	Mean Square	F value	P-value	Prob > F
Model	0.0058	9	0.0006	17.9955	<0.0001	Significant
A-Layer	0.0053	1	0.0053	148.2318	<0.0001	
B-Thread	0.0000	1	0.0000	0.9618	0.3390	
C-Perimeter	0.0000	1	0.0000	0.3882	0.5415	
AB	0.0000	1	0.0000	1.0524	0.3193	
AC	0.0000	1	0.0000	0.0309	0.8625	
BC	0.0000	1	0.0000	0.0736	0.7864	
A²	0.0000	1	0.0001	1.5650	0.2279	
B²	0.0000	1	0.0000	0.9450	0.3446	
C²	0.0000	1	0.0000	0.0170	0.8977	
Residual	0.0006	17	0.0000			
Cor Total	0.0064	26				
R-Squared (R²)	0.905007					
Adj R-Squared (Adj R²)	0.854716					
Pred R-Squared	0.710069					
Adeq Precision	11.77913					

There are many critical regression coefficient values used by other researchers to determine the validity the statistical models developed using Design of Experiments. These values can be considered either in good condition for prediction or not suitable for prediction purposes. For example, Henika (1982) reported that the regression coefficient must be more than 85% in order to be accepted as satisfactory condition for sensory data collection. However, Joglekar & May (1987) reported in their work that the R^2 value should be a minimum of 80% in order to be considered as a good fit. Myers & Montgomery (2002) stated a general rule that a model with regression coefficient below 70% should not be considered as good fit and the model is considered not suitable to represent the data in the experimental design domain.

From Table 4, the statistical model for blunt trauma depth ($\sqrt{\beta}$) prediction can be derived as follows.

$$\sqrt{\beta} = (0.2695) - (0.0070A) + (0.0002B \times 0.0009C) - (5.3381 \times 10^{-06}AB) + (0.0001AC) + (1.6891 \times 10^{-05}BC) + (0.0001A^2) - (1.1031 \times 10^{-06}B^2) - (0.0024C^2) \quad (1)$$

Based from Equation 1, the blunt trauma depth relationship with the significant factors, fabric layers (A) and thread strength (B), can be shown in Figure 4. From this 3D graph, any variation within the parameter range of the fabric layers or thread selection can be used in the prediction of blunt trauma depth of the Kevlar Type 802F stitched panel.

6. CONCLUSION

The effect of stitching parameters on the ballistic resistance capability (in terms of blunt trauma depth) of Kevlar Type 802F has been studied. The ballistic test results suggest that stitching does play a contributing influence to the ballistic resistance of multiple plies of fabrics. Although the number of fabric plies is generally accepted as the main influence in reducing blunt trauma depth, the possibility of using stitching parameters such as stitching pattern and suitable stitching thread materials can further enhance the performance whilst reducing the manufacturing cost. It can be noted that stitching of fabric plies makes the test panel stiffer in comparison with unstitched fabric plies. Although this study is not conclusive as there are other parameters which was not included in the design (needle

diameter, stitch type other than lock-stitch, etc.) that may contribute to the performance, the statistical model can be used as an initial reference in the soft body armour manufacturing process.

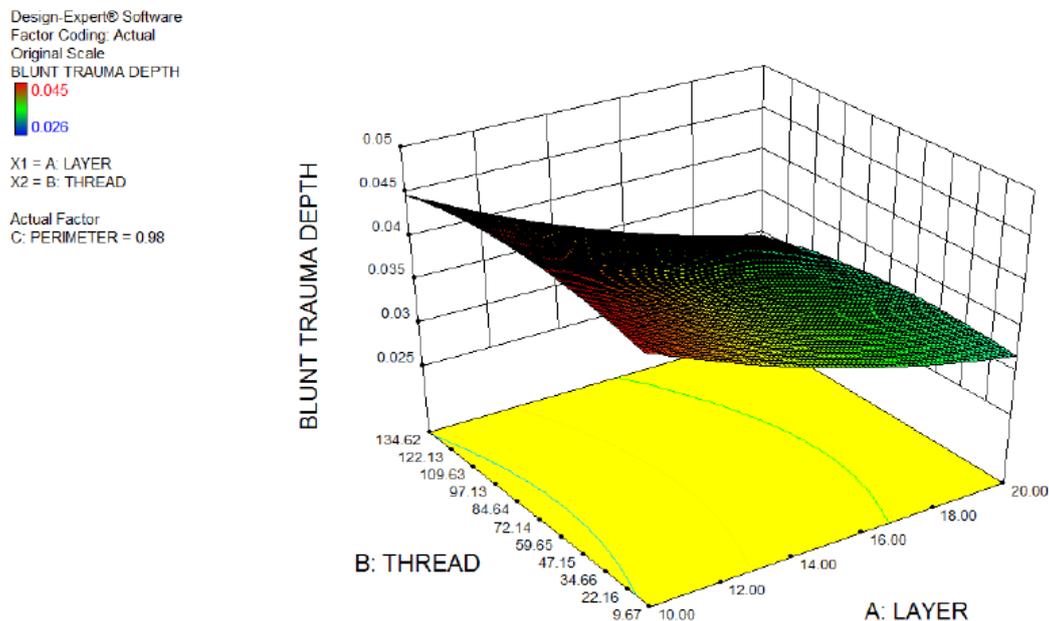


Figure 4: 3D surface representation on effect of numbers of layer and thread strength on blunt trauma depth.

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IDENTIFICATION OF WELDING DEFECTS: A CASE STUDY BASED ON DATA FROM 2010 TO 2012

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ABSTRACT

The welding technique is extensively used for applications such as fabrication and repair work. There is a great variety of welding defects. Some of the defects are considered as one of the factors for some of the catastrophic failures in machineries. In this study, X-ray images captured by the STRIDE's non-destructive testing (NDT) team between 2010 and 2012 are studied to identify the most prominent types of defects. A total of 376 results were produced in this period, with 295 of cases being accepted and the rest being rejected based on severity of the defects. Porosity is identified as the most prominent type of defect, followed by slag inclusion and crack. The cases with two types of defect, such as porosity and slag inclusion, recorded the third highest number of cases.

Keywords: Welding defects; radiographic testing (RT); X-ray images.

1. INTRODUCTION

The welding technique is extensively used in the military for applications such as fabrication and repair work. Welding is a fabrication or sculptural process that joins materials, usually metals or thermoplastics, by causing fusion, which is distinct from lower temperature metal-joining techniques, such as brazing and soldering, which do not melt the base metal (Farmweld, 2010). The welding is normally inspected by using non-destructive testing (NDT), such as radiographic testing (RT) and ultrasonic testing (UT). RT is an appropriate technique for testing welded joints that can be accessed from both sides, with the exception of double-wall signal image techniques used on some pipe. Even with the invention of online digital radiography using sensitive fluorescent plates, offline analogue radiography using films still has its own benefits and applications (Ngon & Toan, 2015). Furthermore, RT is widely used for the welding inspection of hull plating of naval ships. The options for RT include not only film, but with recent technologies advantages, it is now possible to meet a wide range of NDT inspection applications with digital solutions that are reliable and cost effective (Kang *et al.*, 2011).

In this study, X-ray images captured by the STRIDE's non-destructive testing (NDT) team between 2010 and 2012 are studied to identify the most prominent types of defects. This team has been approved by the Atomic Energy Licensing Board (AELB), which is the agency responsible for governing and implementing rules and regulations related on the usage of radioactive material and equipment in Malaysia. This team is equipped with relevant equipment and expertise to perform radiographic work and analysis on X-ray images. At the moment, this team is providing services to local defence industries,

particularly for naval applications, and intend to expand their capabilities and involvement to other military services as well.

2. WELDING DEFECTS

A welding defect is any flaw that compromises the usefulness of a welded joint or area. There is a great variety of welding defects. Some of the defects are considered as one of the factors for catastrophic failure in case of deficiency of corrective measure. Welding defects and their acceptable limits are specified in AWS D1.1: Structural Welding Code - Steel (AWS, 2007). Identifying internal weld defects, such as porosities, longitudinal and transverse cracks, confined slags, and lack of proper fusion, in radiography films is an important objective of welding inspectors. Misinterpretation of radiography films by inspectors, because of factors such as vision error, lack of proper quality of films and fatigue, can cause huge errors in industrial projects. To this end, radiographic image processing has been increasingly used help to eliminate these errors by improving the image quality and making the analysis process easier (Nacereddine *et al.*, 2007; Madani *et al.*, 2015).

A crack is a welding defect generated by a local rupture causing discontinuities on the weld in the metal or on the adjacent heat affected zone when a frozen weld is solidified. Cracks are considered as one of the most serious defects of welded joints and should be removed completely before putting the product into use. Cracks may appear on the surface of the weld, inside the weld and heat affected zones (Farmweld, 2010). Cracks, with different natures, positions and shapes, can be microstructure cracks or cracking of crude cracks. Crude cracks can cause destruction of the structure in working. Microscopic cracks that occur in the process of structural work will spread gradually forming crude cracks (TWI, 2006; Farmweld, 2010). Depending on the cause, location and shape, cracks generally have several categories, such as longitudinal, transverse, branching and crater cracks, as shown in Figure 1 (Ngon & Toan, 2015). X-ray images of cracks, slag inclusion and internal concavity are shown in Figure 2 (Al-Hameed *et al.*, 2013).

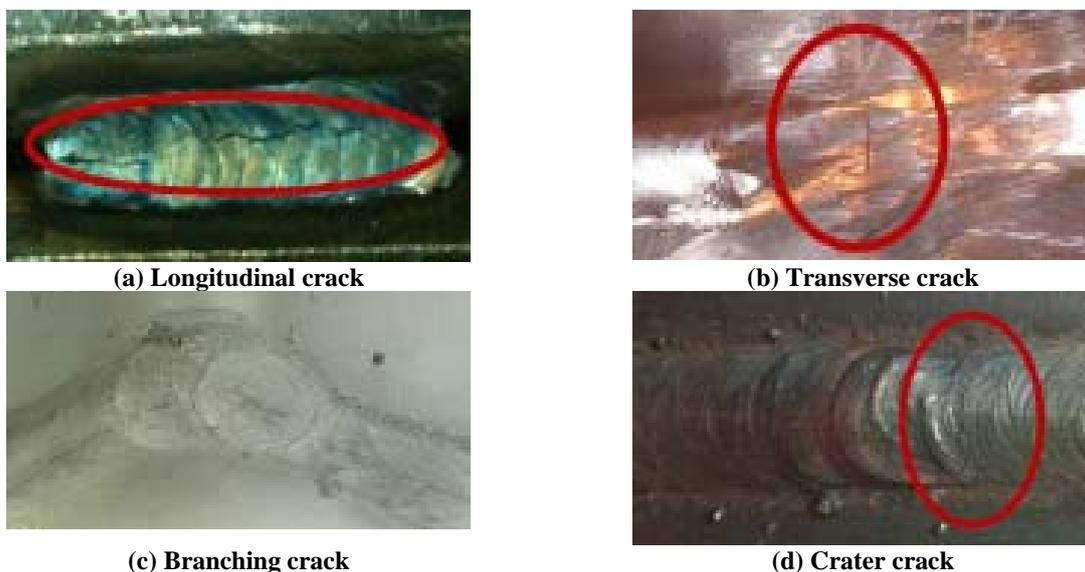


Figure 1: Shapes and locations of different types of cracks.
(Source: Ngon & Toan, 2015)

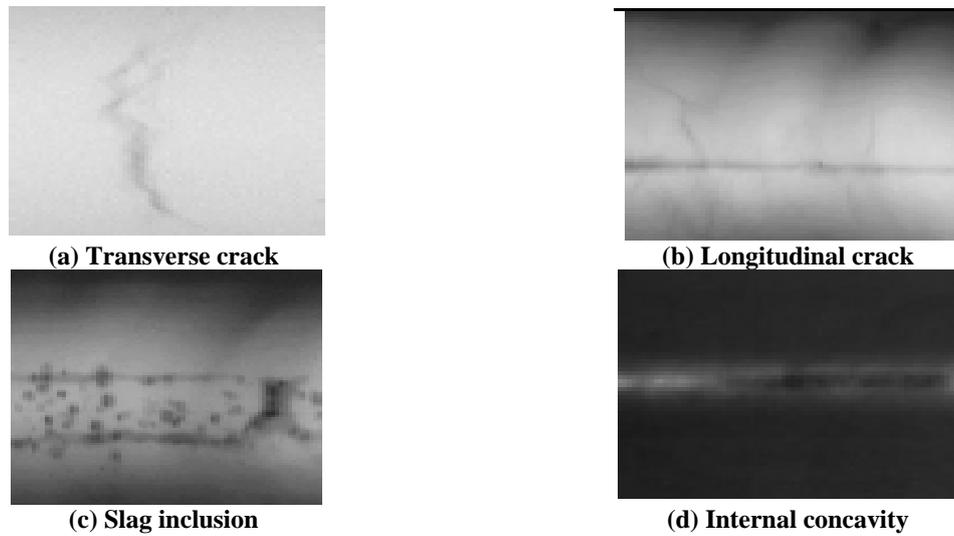


Figure 2: X-ray images of typical welding defects.
 (Source: Al-Hameed *et al.*, 2013)

Internal weld defects, such as porosities (gas voids), slags or slag inclusion, and weld discontinuities, are common welding defects, as shown in Figure 3 (Didžiokas *et al.*, 2008). The X-ray images of the welded joints in the figure show the characteristic defects that occur in welds (Al-Hameed *et al.*, 2013). Slag intrusions occurring in the welded connections can be sporadic or located in a chain (Figure 3(a)). Welding discontinuities (Figure 3(b)) can be located between the layers and along the root of the welding bead. Porosities (Figure 3(c)) can be sporadic, linear or scattered (Didžiokas *et al.*, 2008).

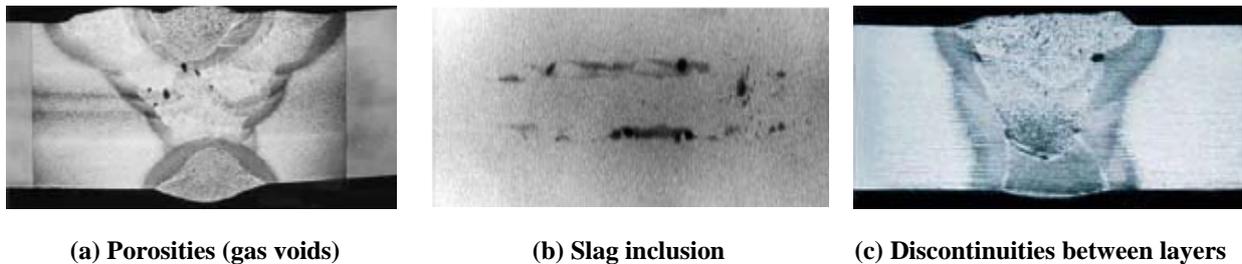


Figure 3: X-ray images of internal weld defects.
 (Source: Didžiokas *et al.*, 2008)

3. METHODOLOGY

The RT method was chosen to determine the internal defects of the welds. The procedure of this method is defined in AWS (2007). The tests were conducted by STRIDE’s NDT team using an X-ray machine (model: ERESO, max. 300 kV). The X-rays that pass through the metal weaken because they are absorbed by the metal’s atomic grating. The weakening process of the X-rays depends on the thickness of the welded area, and its physical and chemical qualities. After passing the welded area, the X-rays of different intensities are recorded on the X-ray films situated on the opposite side of the welded area as

shown in Figure 4. The X-ray films used were Agfa industrial type. Darker spots appear on the film in the defected areas as shown in Figure 5. The defects recorded on the X-ray film are identified according to their type and geometrical parameters (length, width and distance from the weld middle line).

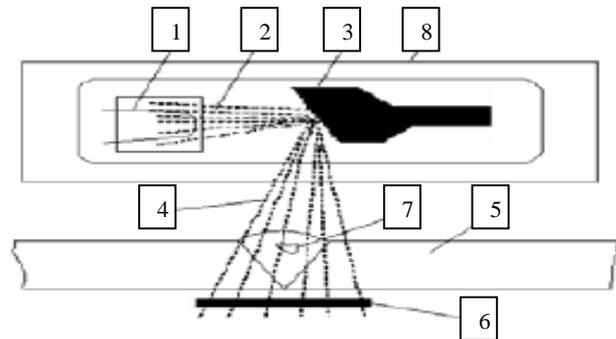


Figure 4: The setup for detecting the internal weld defects.
1–cathode, 2–electron flux, 3–anode, 4–X-ray, 5–the X-ray metal (specimen), 6–X-film, 7–defect, 8–lead case
 (Source: Ngon & Toan, 2015)

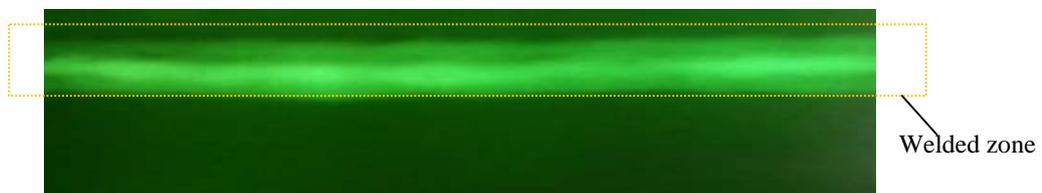


Figure 5: An example of an X-ray image of a defect area.

An X-ray Report (XR) is prepared by STRIDE’s NDT team based on the interpretation on the X-ray images. In this report, the type of the welding defect and size will be stated. In addition, whether the defect is within the acceptance limit will be stated as well. If the defect is not within the acceptance limit, then recommendation for rework will be mentioned in the report. Therefore, the communication between STRIDE’s NDT team and Royal Malaysian Navy (RMN) engineering officers via their repair vendor will be always available to ensure that the quality of the welded area meets the requirements of AWS (2007). The X-ray films will be kept by STRIDE’s NDT team for future reference. In this study, the X-ray reports and their X-ray images between 2010 and 2012 are studied to identify the most prominent types of defects.

4. RESULTS AND DISCUSSION

Table 1 shows the types of welding defects that have been detected using RT technique based on the X-ray images captured during image interpretation. There are six types of defects, including lack of fusion, which was found together with other types of defects, such as slag inclusion, porosity and crack. Some of the X-ray images were found with single type of defect, while others with two types of defects, as listed in Table 1.

Table 1: The grouping of X-ray images.

X-ray images with single type of defect	X-ray images with two types of defects
Porosity – P Slag inclusion – SI Crack – C Lack of penetration – LP Undercut – UC	Porosity and Slag inclusion – P & SI Crack and Porosity – C & P Crack and Slag inclusion – C & SI Lack of fusion and Slag inclusion – LF & SI Lack of fusion and Porosity – LF & P Lack of fusion and Crack – LF & C Lack of penetration and Slag inclusion – LP & SI Undercut and Slag inclusion – UC & SI

Figure 6 shows the common types of welding defects that have been detected. The most common defects were porosities, slag inclusions and cracks. Some of the X-ray images showed more than a single type of defect, in particular porosity and slag inclusion. The other types of defects, such as porosity and slag inclusion; crack and porosity; crack and slag inclusion; lack of fusion and slag inclusion; and lack of fusion and undercut recorded less number of findings.

For the period of 2010 to 2012, a total of 376 results were produced, with 295 cases being accepted and the rest being rejected based on severity of the defects as per stated in AWS (2007). Porosity has been identified as the most prominent type of defect, followed by slag inclusion and crack. The cases with two types of defect, such as porosity and slag inclusion, recorded the third highest number of cases. The most number of cases were recorded in 2012, followed by 2011 and 2010. Basically, the number of cases depends on the number of requests received from the RMN for that particular year.

5. CONCLUSION

Based on the studies on X-ray images that were captured between 2010 to 2012, we were able to identify the most prominent types of welding defects related to welding activities on the hull section of naval ships. Nevertheless, the X-ray technique alone is not suitable to determine the size and exact location of the defect, which normally depends on the type of the defect and the geometrical dimension of the defect. To improve the image processing of X-ray images, new approaches are needed, such as film scanner and specific analysis software, to improve the radiographic work in general.

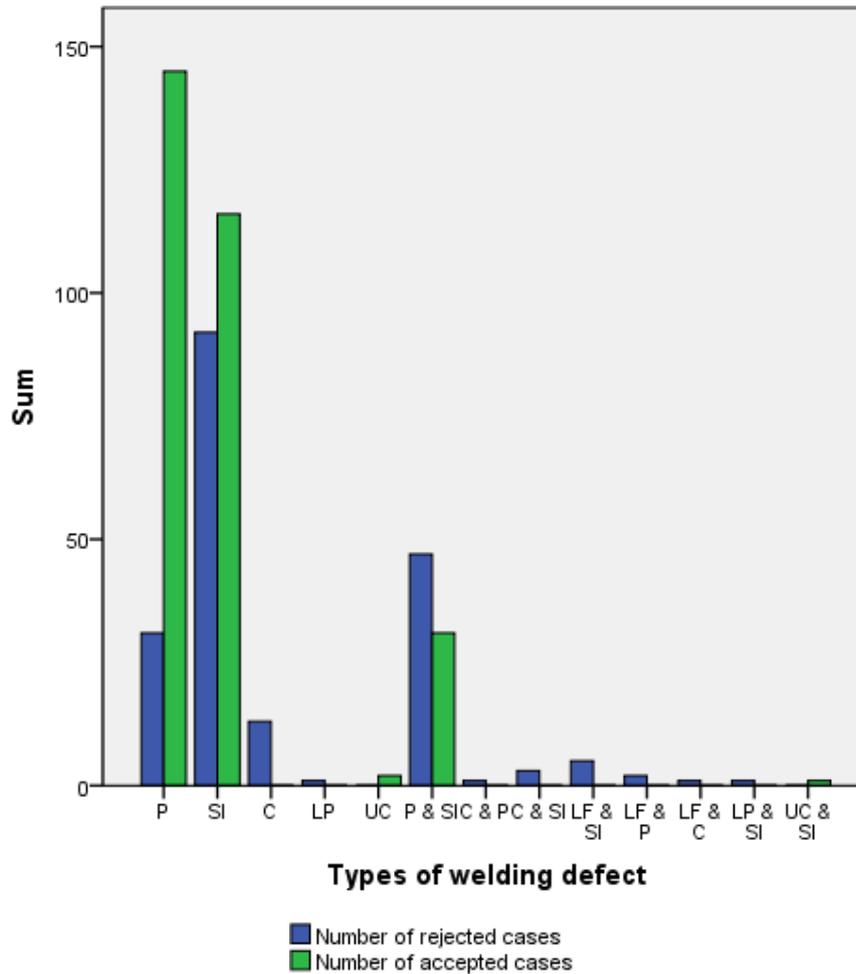


Figure 6: Recorded numbers of welding defects.

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WAVE ENERGY DISSIPATION LABORATORY MODELLING OF SUBMERGED BREAKWATER FOR SHORELINE EROSION CONTROL

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ABSTRACT

Submerged breakwaters provide beach protection by dissipating the wave energy for shoreline erosion abatement without destroying beach aesthetics. The geometrical characteristics of the structural design of the submerged breakwater determine its stability. Wave magnitude propagation in the actual physical sea state conditions, particularly wave period, height and depth, are vital factors that dictate wave transformation. Laboratory experiments in a controlled systematic programme are modelled in the condition of monochromatic unidirectional wave to present coefficient of transmission (C_t) as the reference index of breakwater wave energy dissipation for the effect of wave period, significant incident wave height and structural narrow crest width. The test results indicate high wave suppression and structural friction to wave motion, where C_t values tend to decrease with the increment value of relative wave period, significant incident wave height and relative narrow crest width.

Keywords: *Submerged breakwater; wave dissipation; coefficient of transmission; hydrodynamic physical modelling; physical sea state conditions.*

1. INTRODUCTION

The National Coastal Erosion Inventory Study indicated that 1,415 km (29.41%) of Malaysia's coastline is classified as eroded. Coastal erosion can be due to natural processes of coastal sediment transport dynamics not in equilibrium or man-made intervention such as structures built along the coastline that disturb the dynamic equilibrium of the coastal sediment transport. The consequence is the loss or threat of damages of existing facilities, such as residential areas, infrastructure, valuable land, natural habitats and vegetation. The damages could also involve national defence infrastructure, such as naval bases and ports (NCVI, 2006). In a recent event, the combination of coastal tidal phenomena and wind storm triggered the evacuation of residents by the sea side in Port Kelang, Selangor and Kuala Muda, Kedah. The dynamic wave drag energy overflowed the coastal revetment, thus flooding the infrastructure and causing severe beach erosion (BH, 2016).

In the past two decades, submerged breakwaters have been increasingly used in coastal management for protecting beaches from erosion. One of the advantages of this structure is that the coastal protective functionality can be fulfilled without spoiling the landscape. This is increasingly important in recreational and residential coastal developments. In addition, submerged breakwaters absorb some of the incoming wave energy by causing the wave to break prematurely, thus diminishing the transmitted wave energy. Increasing interest in utilising submerged breakwaters and artificial reefs for shoreline stabilisation requires accurate predictive models and relationships for wave attenuation (Arnouil, 2008). The effects of wave period, significant incident wave height and narrow structural crest width of breakwater at different water depths were analysed by Ahmad *et al.* (2014) for different physical sea state hydrodynamic

conditions, and parameters of coastal behaviour and structural type. It appears that wave energy dissipates in the lee of a submerged breakwater after transmission by the process of friction, and thus breaks and reflects. Chatchawin *et al.* (2015), in the modelling of seadome marine structure, found that wave energy reduction of 50% occurs in the interaction of seadome to the generated incident wave. The working features of innovative coastal defence structures that can dissipate the energy of incoming waves by the action of large-scale bottom unevennesses (rigid blades covering the lower half of the water depth) were investigated Lorenzoni *et al.* (2010) by means of a laboratory experimental campaign to characterise the ability of the structures to efficiently reduce the wave height with a minimal change in the mean water superelevations.

However, most studies on wave propagation over submerged breakwaters are based on the plane bed located the near shore (Turner *et al.*, 2005). This paper presents a narrow crest submerged breakwater, known as Wave Breaker Coral Restorer (WABCORE), developed by the National Hydraulic Research Institute of Malaysia (NAHRIM). The interlocking character of WABCORE, achieved by easily performing simple trapezoidal design of narrow crest, allows a module unit of WABCORE to be configured between a single and more layers that represent bigger dimension in height or width subject to hydrodynamic character and wave condition of each different places to produce significant results on transmission coefficient (Fauzi *et al.*, 2013). This study will report on the findings of tests conducted to evaluate the ability of WABCORE for wave suppression and structural friction to wave motion, based on actual physical sea state conditions for parameters of relative wave period, significant incident wave height and relative narrow crest width.

2. MATERIAL AND METHOD

2.1 Experimental Setup

The hydrodynamic physical modelling was carried out in the laboratory of NAHRIM. Generally, the largest possible scale is selected, subject to the test facility's capability. Smaller scales might affect the repeatability by which the small scale wave can be reproduced in the laboratory. Thus, flume size and wave paddle capability are important parameters before any scale effect selected that assure the dominant forces are well represented. The necessary scale of model is chosen in such a manner that all important wave conditions and structural parameters are reproduced to an adequate measuring accuracy. A large model scale is often necessary for the accurate modelling of wave loading and structural response (HYDRALAB, 2007). Therefore, for this particular experiment, Froude's Law with scale effect of 1:4 was determined. This research was embarked in 2D flume with 100.0 m length, 1.5 m width and 2.0 m height. Regular monochromatic wave forces produce consistent wave magnitude and amplitude that were controlled by the sensor gauge at the wave paddle, which were then calibrated in the flume before the model was built to ensure that the incident wave measurements recorded by array of wave probe are not corrupted by reflected waves. Three wave probes, labelled as WP1, WP2 and WP3 respectively, were installed at 25, 30 and 32 m down the flume from the wave paddle, whilst WP4 and WP5 were set at 2 and 5 m after the centre line of WABCORE, as shown in Figure 1. For the regular wave calibrations, a wave train of 50 fully developed waves were run and the wave heights derived from the average of each max and min, peak and trough pair in each single wave were recorded.

2.2 WABCORE Construction

Normal concrete admixture was used for the casting of WABCORE model and it was left for curing purposes in the laboratory for seven days to achieve maturity as specified in BS 8110. The method of similarity was followed thoroughly with the approved designed of scale effect of 1:4 and model construction in geometric similarity whilst wave propagation complied to kinematic and dynamic

similarities. It was decided that the WABCORE model would be constructed in the arrangement of a four-layer trapezoidal shape, with wide bottom and narrow hat of 4, 3, 2 and 1 units respectively from the bottom, second and third middle layers followed by the top hat. Figure 2 shows the progress of the construction of WABCORE from the fabrication of steel mould, concrete curing and narrow hat arrangement.

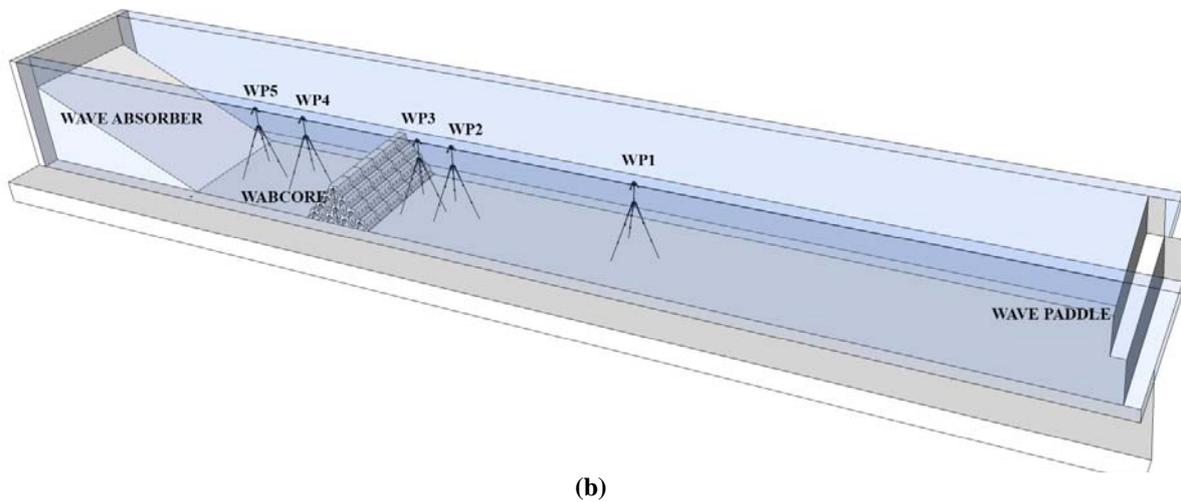
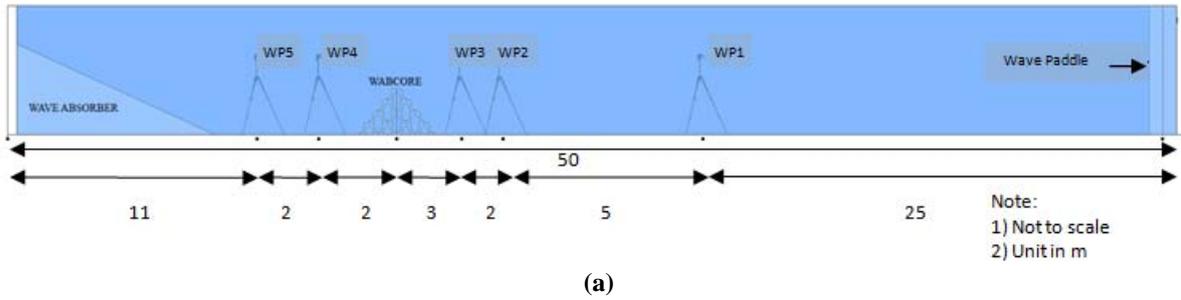


Figure 1: (a) Cross-sectional and (b) aerial views of the experimental setup.



Figure 2: Progress of construction of WABCORE: (a) Fabrication of steel mould. (b) Concrete curing. (c) narrow hat arrangement.

2.3 Test Parameters

Primer wave data were collected earlier in the actual prototype deployment site at Pulau Tinggi, Johor by using of an acoustic Doppler current profiler (ADCP). This instrument was deployed for 14 days to record the complete cycle of tidal phenomenon. The hydrodynamic output was analysed and the test parameters were decided as shown in Table 1.

Table 1: Test parameters used in this study.

Wave Type	Monochromatic
Wave Period, T_p (s)	1.50, 1.75, 2.00, 2.50
Wave Height, H_i (m)	0.10, 0.15, 0.20, 0.25
Water Depth, d (m)	0.50, 0.75, 1.00, 1.25

3. RESULTS AND DISCUSSION

The coefficient of transmission (C_t) is defined as the ratio of the transmitted wave height (H_t) shoreward of the breakwater to the incident wave height (H_i) seaward of the breakwater, $C_t = H_t/H_i$ (Faridah *et al.*, 2007). The greater the value of C_t , the worse effect of wave attenuation to the leeside of the breakwater, and vice versa. Four water depths were used in the experiment, $d = 0.50, 0.75, 1.00$ and 1.25 m, corresponding to relative water depths $h/d = 1.00, 0.67, 0.50$ and 0.40 at the constant structural height, $h = 0.50$ m.

3.1 Effect of Relative Wave Period on Transmission Coefficient

Relative wave period is presented by H_s/L , where H_s is the significant wave height and L is the wavelength. The transmission coefficient is known affected by the wave period, which controls the wavelength and type of wave. High wave period results into long wavelength, which leads to lower transmission because longer waves have more potential of just passing over the structure. At water depth of 0.5 m, $h/d = 1.00$, the C_t value is 0.6856 . C_t tends to become smaller at the increment of value H_s/L , which indicates shorter wave period. Hence, H_s/L of 0.0889 gave the smallest value of C_t of 0.4451 , as indicated in Figure 3. At deeper water depth (0.75 m), $h/d = 0.67$ shows larger value of C_t at 0.4842 , which represents more than 8% wave energy increase. C_t tends to escalate at h/d of 0.50 and 0.40 , with values of 0.6607 and 0.7203 respectively. The effect of wave period is dominant when water depth is at the structural crest level and shorter wave period allows friction of wave length to the crest surface.

3.2 Effect of Significant Incident Wave Height on Transmission Coefficient.

The effect of significant incident wave height was quantified by significant incident wave height to water depth ratio (H_s/d). Designed wave height were induced by setting the amplitude of the wave generator setting parameter. The results in Figure 4 show that at the minimum water depth and low amplitude of wave height, C_t is extremely high, which indicates low impact of wave transformation. Nevertheless, at minimum water depth and bigger wave height, C_t becoming smaller, proving that wave height is an important factor for wave attenuation. At the relative water depth, $h/d=1.00$ and incident wave height generated at 0.25 m, the value of C_t turns smallest at 0.4451 , showing that the wave dissipated

tremendously. The other relative water depths $h/d=0.67$, 0.50 and 0.40 also indicate similar trend of plotted C_t .

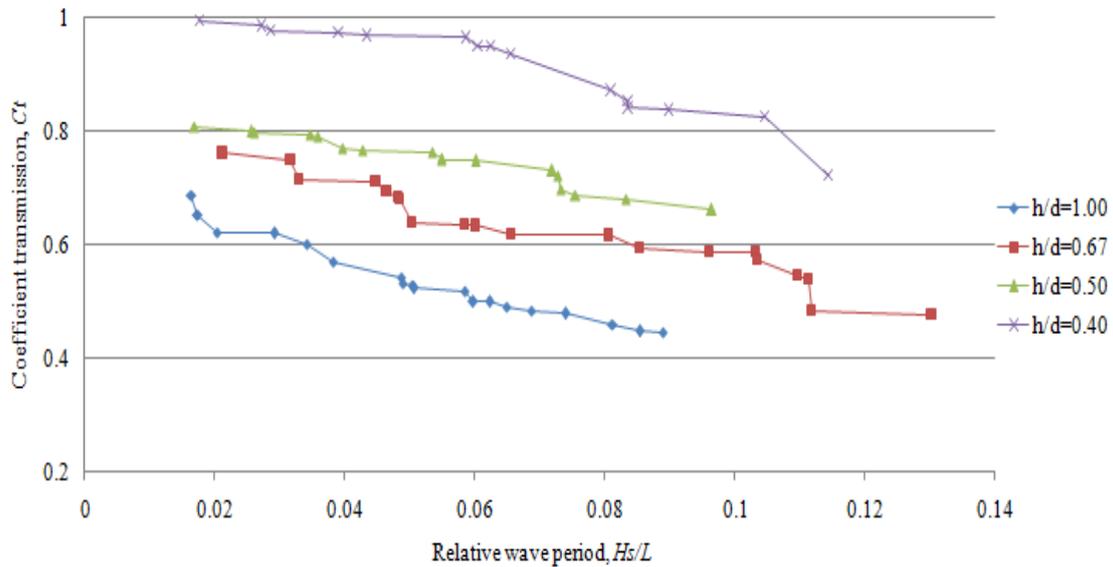


Figure 3: Effect of relative wave period on transmission coefficient.

3.3 Effect of Relative Structural Crest Width on Transmission Coefficient

The reduction in transmission occurs because the crest span starts to provide friction in overtopping wave that is defined by crest width to wave length ratio (C_w/L). Shorter wavelengths allows for maximum interaction of wave magnitude to the crest width as compared to longer wavelengths that just allow the wave to pass over the crest with less interaction (Teh, 2014). Figure 5 shows similar trends for all h/d , whereby the C_t diminishes with the increasing value of C_w/L . This proves that the breakwater performs efficiently in the hydrodynamic condition dominated by shorter wavelength. C_w remains consistent throughout the experiment at 0.10 m while the values of L were based on designed T_p . At $h/d=1.00$, the C_t value was 0.4451 at the shortest of L . An increment of 60% wave energy occurred at $h/d=0.40$ whereby C_t was 0.7203. Alternatively, lower wave transmission can also be attained by increasing the crest width of the breakwater, which allows structural surface interaction to the high amplitude of wave and wavelength.

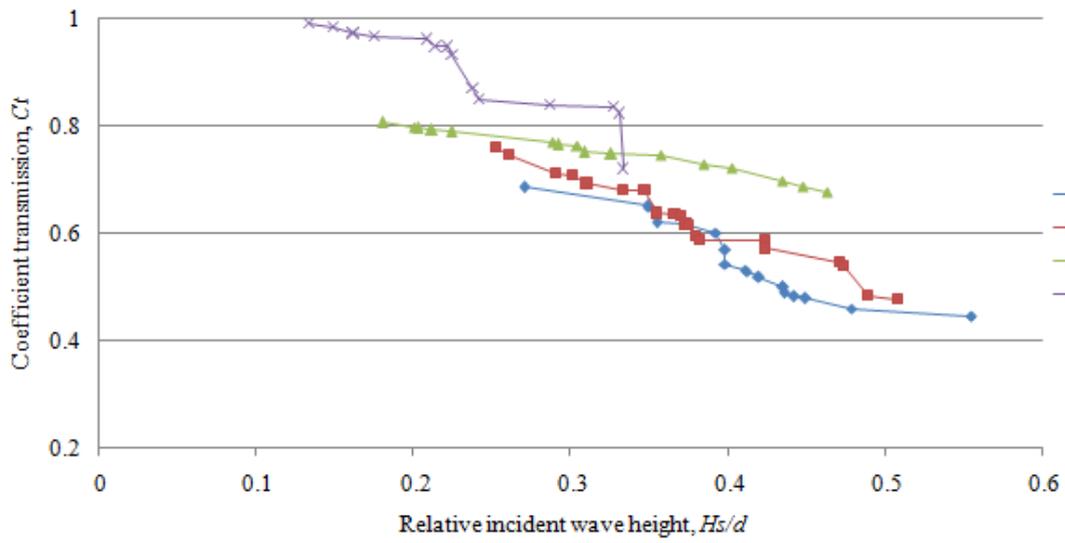


Figure 4: Effect of significant incident wave height on transmission coefficient.

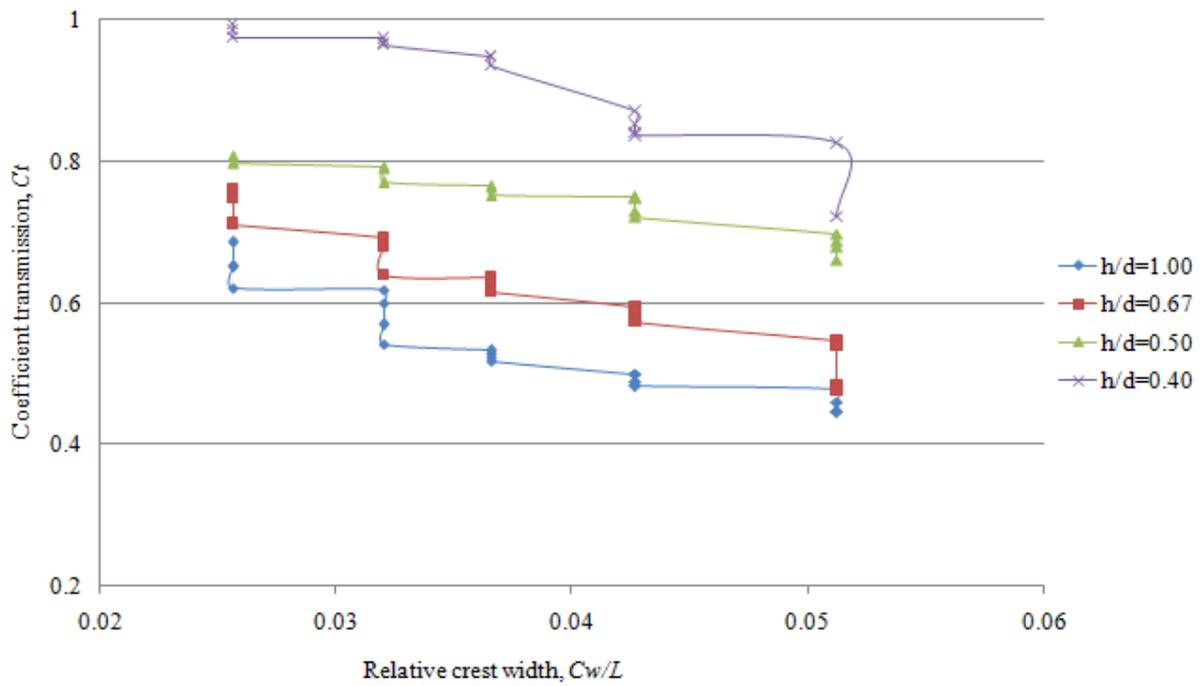


Figure 5: Effect of relative crest width on transmission coefficient.

4. CONCLUSION

The C_t plots for WABCORE shows tremendous performance for wave transmission. Subject to different sea state conditions, such as water depth, wave period, incident wave height and wave length, the indicated high wave suppression and capability of WABCORE allows for wave breaking and structural friction to wave motion. Physical sea state conditions, such as high amplitude of incident wave height, structural height, crest width span and wave period, show correlation to the performance of WABCORE. The wave energy dissipated by 65% in the test limit of relative water depth, $0.40 \leq h/d \leq 1.00$; relative wave period, $0.0164 \leq H_{si}/L \leq 0.1303$; incident wave height, $0.1338 \leq H_s/d \leq 0.5547$; and crest width, $0.0256 \leq Cw/L \leq 0.0512$ for regular monochromatic wave. The attenuation of wave energy has given promising potential of submerged breakwater to protect vulnerable coastlines from erosion, and thus protect infrastructure and residents in the shoreline settlement. This research could be a flagship of National Blue Ocean Strategy across various ministries, such as Ministry of Defence, Ministry of Natural Resources and Environment, Ministry of Agriculture as well as Ministry of Science, Technology and Innovation to collaborate on research in coastal conservation.

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EXTRACTION OF SILVER NANO PARTICLE FROM X-RAY WASTE USING PROTEOLYTIC ENZYME

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ABSTRACT

Alkaline protease from Bacillus firmus shows enzymatic hydrolysis of gelatin from waste X-ray films and produces silver nano-particles. At the end of the treatment, gelatin layer was completely removed leaving the polyester film clean and silver was recovered in the hydrolyte, both of which can be reused. Silver is linked to gelatin in the emulsion layer, it is possible to break the same and release the silver using proteolytic enzymes. Around 18-20 % of the world's silver needs are supplied by recycling photograph waste. Various parameters on silver removal from the film such as pH, temperature, enzyme concentration, time etc. were studied. Gelatin hydrolysis was monitored by measuring increase in turbidity, which was accompanied by release of protein and hydroxyproline. Gelatin layer was stripped completely within 6 minutes with 1.35 U/ml of protease at 40°C at pH 10. Rate of gelatin hydrolysis increased with increased protease concentration. The enzyme could be effectively reused for four cycles of gelatin hydrolysis. Silver in hydrolysis was around 3.87 % (w/w) based on total weight of sludge. The waste X-ray/photographic films contain 1.5 - 2 % (w/w) black metallic silver which is recovered and reused.

Keywords: *Bacillus firmus; nano-particles; x-ray film; hydrolysis.*

1. INTRODUCTION

Screening of alkaliphiles from naturally occurring habitats in different parts of the world expected to result in isolation of new protease producing organisms potentially useful for many applications. Alkaline proteases have numerous applications in food industries, silver recovery from x-ray films and several bioremediation processes (Fujiwara *et al.*, 1991). One of the most important and noteworthy features of many alkaliphiles is their ability to modulate their environment. They can convert any neutral or high alkaline medium in their favor to optimize external pH for their growth (Rao & Narasu, 2007). Proteases of the subtilisin groups are used in the pharmaceutical industry for the treatment of burns and wounds. The involvement of proteases in the life cycle of disease causing organisms has led them to become a potential target for developing therapeutic agents against fatal diseases, such as cancer and AIDS (Rao *et al.*, 1998).

The integration between nanotechnologies and biology and medicine is expected to bring evolutionary advances in diagnostics and therapeutics, molecular biology and bioengineering. Recent discoveries resulted in the production of functional nanoparticles, covalently bound to biological molecules like peptides, proteins and nucleic acid (Whaley *et al.*, 2000). Nowadays, biocompatible peptide-covered nanoparticles are part of specific sensor systems used in natural sciences applied in molecular recognition-based immunoassay (Parak *et al.*, 2003). Nano-particles are expected to be the basis for the

future creation of "intelligent packaging" in the food industry (Berekaa, 2015). These new strategies will grant the manufacture of packages with gas transfer through the package layers and giving information on the status of frozen foods (Krumovet *al.*, 2009). The use of an alkaline protease to decompose the gelatinous coating of x-ray films, from which silver is recovered. Proteases are also useful and important components in bio-pharmaceutical products such as contact-lens enzyme cleaners and enzymatic debriders (Rao & Narasu, 2007).

2. MATERIAL AND METHODS

Three soil samples were collected from the Mahatma Gandhi Mission's college campus, Nanded, Maharashtra, India. We collected the soil from different sources such as soil from roots of rose plant, soil from iron sample and soil from college ground. This soil is used as source of microorganism. This source is used for isolation & screening process of microorganism.

The following steps were followed for isolation & screening process:

Enrichment: For enrichment of bacteria high protein broths were prepared & autoclaved at 10 lbs for 30 min. After cooling of broth 1 gm soil is inoculated in each sample. This sample was kept for enrichment in incubator at 37°C for 48 h (Rao & Narasu, 2007). High protein media consisting of: Corn meal, 1.5g; soya meal, 1.5g; Na₂HPO₄, 0.02 g; KH₂PO₄, 0.012g; MgCl₂, 0.01g; CaCl₂, 0.012g; Na₂CO₃, 0.012g; glucose, 0.036 g; and distilled water, 100ml.

Isolation: For isolation of microorganism, the high protein media were prepared & autoclaved at 10 lbs pressure for 30 min. The autoclaved media is poured in autoclaved petriplates in aseptic condition & allowed it to solidify. After solidification 0.1ml of enriched sample was inoculated and spreaded on media plates. These plates were incubated for 24 h at 37°C. The plates were then checked for colonies of bacteria (Rao & Narasu, 2007).

Screening for proteolytic bacteria: The bacterial isolate then inoculated on to the casein and gelatin agar plates at 37°C for 24 h. The media consist of glucose, 1 gm; Yeast extract 0.5g, Tryptone, 0.5 g, trisodium citrate, 0.44 g; CaCl₂, 20 mM; agar, 2 g; and distilled water, 100ml.

The clear zone of hydrolysis gave an indication of proteolytic microorganisms. The colony showing clear zone are maintain on nutrient agar slants and their colony characterization and basic biochemical tests were carried out. Only the soil sample collected from the roots of Rose plant shows the clear zone of hydrolysis which gave an indication of proteolytic microorganisms. So, for further morphological and biochemical characterization the colonies from Rose root soil sample is used. Now isolated colonies are used for characterization of microorganism.

Characterization of selected isolate: Morphological and physiological properties of selected isolate ATCC 14575 were investigated according to the methods described in "Bergey's Manual of Systematic Bacteriology 1986".

Production media optimization: Yeast extract casein broth is used as production media. Before production of an enzyme, the Yeast extract casein media is optimized by using Plackett-Burman (PB) design.

Screening of medium components and operating conditions by Plackett-Burman (PB) design is an efficient screening design when main effects of the medium components are to be considered. PB design offers a good and fast screening procedure and mathematically computes the significance of a

large number of factors in one experiment, which is time saving and gives the effects of change in more than one factors in single experiment. In this work, PB design in 12 experimental run was carried out to evaluate the effect of 6 factors of medium components and operating conditions on ALKALINE PROTEASE production. All the factors are prepared at two levels "-1" for low level and "+1" for high level. The factors (% W/V), such as - glucose (X1), yeast extract (X2), casein(X3), KH₂PO₄ (X4), K₂HPO₄ (X5), MgSO₄ (X6) (Baskaret *al.*, 2011) were studied. The actual values of the variables at low level (-1) and high level (+10) are given in Table 4.

Enzyme purification: The partial purification is carried out of the three sets of production Medias are as follows:

Protein extraction method: After two days of incubation, the flask was removed from shaking incubator and subjected to filtration to remove the cell debris and other impurities. Then, filter through Whatmann filter paper no.1, collect the filtrate and centrifuge it at 10,000 rpm for 15 min at 4°C (Najafiet 2005).

Protein Precipitation Method:For ammonium sulphate precipitation, 100 ml of supernatant used for precipitation with 70 % ammonium sulphate. All steps carried out at 40°C. The resulting precipitate was collected by centrifugation at 15,000g for 30 min at 40°C. The precipitate dissolved in 20mm TrisHCL (pH 9) and used for gel filtration chromatography or dialysis.

Purification of extracellular enzyme: Principle of dialysis: Dialysis is the method used to separate dissolved molecule on the basis of the molecular size. the technique involves sealing of aqueous solution containing both micro and macromolecules in the porous, this membrane not allow diffusion of macromolecules of weight greater than 10000 dalton, while small molecules diffuse through the pores of membrane. The passage continues until the concentration inside the dialysis tube reaches equilibrium that is achieve after 4-6 h.

Procedure: The membrane of dialysis tube cut in to pieces of conventional size (10-20 cm) and boiled for 10 min in a large volume of 2% w/v NaHCO₃and1NNaOH. Afterwards it was boiled in 1MEDTA (Ethylene diamine tetra acetic acid) for 10 min. The membrane cooled and stored at 40°C. After this the extracted protease enzyme packed in to dialysis membrane and this was suspended in water containing beaker and kept on a magnetic stirrer for 6-8 h. The enzymatically active fraction was finally purified protease enzyme.

Production and Purification: The production and partial purification is carried out of the 3 sets of production Medias are as follows: 250 ml of yeast extract casein media was sterilized in autoclave at 10 lbs pressure for 10 min. After cooling the media, inoculate it with loop full culture of isolated bacteria. This media then kept in shaking incubator at 180 rpm (revolution per minute) at 25 °C for 48 h (Rao & Narasu, 2007).

Assay of enzyme: Proteolytic activity in the culture supernatant was determined by using the spectrophotometric method (Chopra &Mathur, 1985) with slight modification. 1 ml of enzyme solution incubated with 1 ml of 2% casein in phosphate buffer (50 mM, pH 7) at 400 °C for 10 min and the reaction terminated by the addition of 5 ml trichloroacetic acid (5%). After 30 min, the mixture was filtered and 2 ml of filtrate was added to 4 ml 0.1 N NaOH and 0.5 ml diluted Folin-Ciocalteau reagent. Absorbance was then measured at 670 nm. One unit of enzyme activity was defined as the amount of enzyme required to release 1 µg of tyrosine/min under standard conditions (Shankar & More, 2010).

Effect of pH on enzyme activity: The activity of crude protease was measure at different pH values. Enzyme extract (0.1 ml) was pre incubated with 1.9 ml of buffer (phosphate buffer of ph range 5.0 and

6.0; TrisHCL buffer of pH range 7.0 and 8.0; glycine NaOH buffer of pH range 9.0 and 10.0) at 370 °C for 2 hrs ; 1ml of 2.5% casein was added and the mixture was incubated at 370 °C for 30 min. At the end of incubation period , the reaction was arrested by the addition of 2 ml trichloroacetic acid. In addition, enzyme blank was always included. The tubes were allowed to remain at room temperature for 30 min and the solution was filtered through Whatmann filter paper no. 1. The optical densities of the trichloroacetic acid soluble materials were read a 670 nm. (Oliveria AN.,2010)

Effect of temperature on enzyme activity:The activity of crude protease was determined by incubating the reaction mixture at different temperature. The reaction mixture (1ml of casein solution 2.5% + 1.9 ml of glycine NaoH buffer (0.1 M, pH 8) + 0.1 ml of crude enzyme was incubated at different temperatures (200, 300, 400, 500, 600 and 700 °C), for 30 min. At the end of incubation period, the reaction was arrested by the addition of 2ml 5% Tri choloro acetic acid. In addition, enzyme blank was always included. The tubes were allowed to remain at room temperature for 30 minute and the solution was filtered through whatman filter paper no. 1 the optical densities of the trichloroacetic acid soluble materials were read a 670 nm (Oliveria, 2010).

Effect of carbon source: Various carbons sources Dextrose, Fructose, Sucrose, Xylose, Lactose, Maltose, and Mannitol were used in production medium at concentration of 1% w/v to check the effect of carbon source on enzyme production (Bradford, 1976).

Determiration of Alkaline Protease: The activity of Alkaline Protease was determined by using FTIR (Fourier transform infrared spectroscopy) (SHIMADZU) (SHIMADZU, 2016).

Protein Determiration: Protein was determined by Bradford in 1976 and Bovine serum albumin standard (Shanka, 2010)

The Degradation of X ray films and synthesis of silver nanoparticles: The used X ray films was washed with distilled water and impregnated with ethanol, and was cut into 4 x 4 cm pieces after drying in an oven at 400 °C for 30 min. Each of the film was rinsed in series 100 ml stock enzyme extract and the ph of the solution was adjusted to 9. The solution and the films stirred at 500 °C in a water bath until the gelatin layer stripped completely; obtained slurry was then dried in the presence of borax at 11, 000 °C in a furnace. The purity of the silver was determined potentiometrically (Shankar & More, 2010)

Biosynthesis of silver nanoparticles:The sterile X ray films are added to the sterile yeast extract casein media, kept this flask in shaking incubator for 48 h at 250 °C and 180 rpm. After incubation centrifuge the media to remove the cell debris then this sample was added to boiled dialysis bag. Sterile distilled water was added in a beaker then suspends the dialysis bag in to beaker and kept on a magnetic stirrer for 6-8 h. The silver nanoparticles are released in the beakers which are UV-spectrophotometrically analyzed (Sastry, 2003).

3. RESULT & DISCUSSION

3.1 Colony Characters and Biochemical Characterization

The soil samples were collected from a research center. This isolated, bacteria was selected for further studies because of its potential as a good extracellular alkaline protease producer. Table 1 shows colony characteristics of the sample collected. After 2-3 days of incubation, enrichment of microorganism is occurs in broth (Figure 1). The broth appears turbid, and thus, it is concluded that large microbial growth is developed. During the isolation the enriched suspension is spread on

isolation medium having high pH (10). After incubation the isolated colonies appeared on plate (Figure 2). On that plate only those organism having capacity to grow at high pH only that organism grow on isolation medium. On the basis of morphological characterization from the observation the organism is Rod shaped, white in color, motile, Gram positive, etc. With reference to Table 2, Biochemical Characterization Catalase test is +ve due to it catalyses the breakdown of hydrogen peroxide (H_2O_2) with release of oxygen in the form of air bubble, Indole -ve due to the organism not having ability to breakdown tryptophan to indole, casein +ve due to having ability to hydrolyses casein to produce more soluble transparent derivatives, Starch +ve due to organism having ability hydrolyses starch to produce maltose glucose, and dextrin, Gelatin +ve the organism cleaves gelatin to polypeptides and then further degrades polypeptides to amino acids, Nitrate reduction +ve organism to reduce nitrate to nitrites or free nitrogen gas, ugars fermentation(Figure 3), Methyl red +ve (Figure 4). From these morphological and biochemical characterization the organism is *Bacillus firmus*. After fermentation the enzyme is produced into the broth. On the basis of its extracellular nature it is synthesized inside the cell and released into the medium. In Table 1, morphological characters of microorganism is listed. The various biochemical characterization tests were carried out, the results of those tests are listed in Table 2. In Table 3, the results of the sugar tests are listed. All these tests are required to identify the name of microorganism according to Bergey's Manual.



Figure 1: Hydrogen sulphide test.



Figure 2: Sugar fermentation test of dextrose.



Figure 3: Starch hydrolysis test.

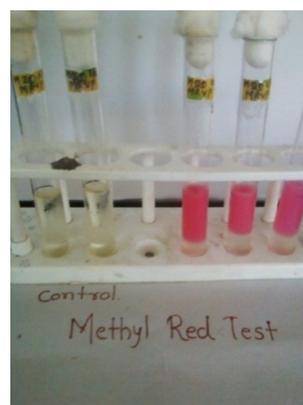


Figure 4: Methyl red test.

Table 1: Colony characters.

Sr. No	Character	Observed character
1	Size	3mm
2	Shape	Circular
3	Color	White
4	Margin	Rhizoidal
5	Elevation	slightly raised
6	Opacity	Opaque
7	Consistency	Sticky
8	Surface	Smooth
9	Mobility	Smooth
10	Grams Nature	Gram positive

Table 2: Biochemical characterization.

Biochemical tests	Positive/ Negative test Indicator
Growth on MacConkey Agar	Negative
Indole Test	Negative
Methyl Red Test	Positive
VogesProskauer Test	Negative
Citrate Utilization	Negative
Gas from Glucose	Negative
H ₂ S Production	Positive
Casein Hydrolysis	Positive
Gelatin Hydrolysis	Positive
Starch Hydrolysis	Positive
Urea Hydrolysis	Negative
Nitrate Reduction	Negative
Catalase Test	Positive
Oxidase test	Positive

Table 3: Sugar test.

Sr. No.	Sugar test	Gas production	Acid production		
			Test tube 1	Test tube 2	Test tube 3
1	Dextrose	Absent	Present	Present	Present
2	Sucrose	Absent	Present	Present	Present
3	Xylose	Absent	Present	Present	Present
4	Lactose	Absent	Absent	Absent	Absent
5	Maltoes	Absent	Present	Present	Present
6	Mannitol	Absent	Present	Present	Present
7	Fructose	Absent	Present	Present	Present

3.2 Effect of Media Component

Media optimization: As shown in Table 4, twelve different Medias were composed to evaluate the effect of 6 factors of medium components and operating conditions on alkaline protease production. All the factors are prepared at two levels "-1" for low level and "+1" for high level. The factors (% W/V), such as glucose (X1), yeast extract (X2), casein(X3), KH₂PO₄ (X4), K₂HPO₄ (X5), MgSO₄(X6), were studied. The actual values of the variables at low level (-1) and high level (+10) are given with results.

Citrate test (Figure 5) (Forbes *et al.*, 2002), Voges-Proskauer test (Figure 6) (Mac, 2000), Starch hydrolysis test (Figure 7) (Clarke & Kirner, 1941; Intuitive Systems, 2016) and hydrogen sulphide test (Figure 8) were performed in the lab, which are indicated in Bergye's manual.

Table 4: Media optimization.

Experiment no	Glucoses	Yeast Extract	Casein	KH ₂ PO ₄	K ₂ HPO ₄	MgSO ₄	Growth on plates
1	+1	-1	+1	+1	-1	-1	Minimum
2	+1	+1	-1	-1	-1	-1	Minimum
3	-1	+1	+1	+1	+1	-1	Minimum
4 (sample 1)	+1	-1	+1	+1	-1	+1	Maximum
5 (sample 2)	+1	+1	-1	-1	+1	-1	Maximum
6	+1	+1	+1	+1	+1	+1	Minimum
7	-1	+1	+1	+1	-1	+1	Minimum
8	-1	-1	+1	+1	+1	-1	Minimum
9	-1	-1	-1	-1	+1	+1	Minimum
10	+1	-1	-1	-1	+1	+1	Minimum
11	-1	+1	-1	-1	-1	+1	Minimum
12	-1	-1	-1	-1	-1	-1	Minimum

Maximum growth on plates indicates: More zone of hydrolysis on plates.

Minimum growth on plates indicates: Less zone of hydrolysis on plates



Figure 5: Citrate utilization test.



Figure 6: Voges-Proskauer test.

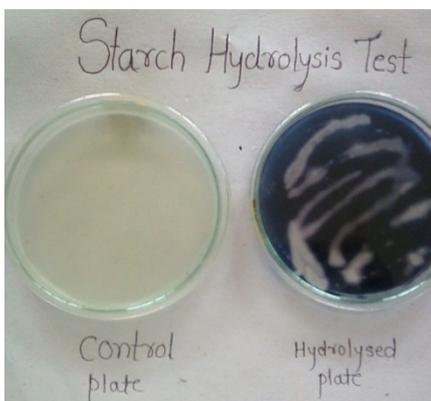


Figure 7: Starch hydrolysis test.

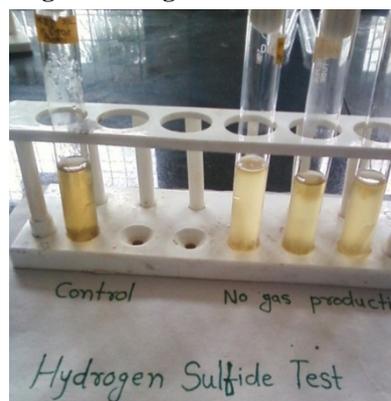


Figure 8: Hydrogen sulphide test.

After isolation, purification & assaying the enzyme various tests were taken which are important for enzyme study:

Effect of temperature and pH: Effect of temperature and pH was studied at fixed protease concentration of 1.35 U ml^{-1} . Gelatin hydrolysis increased with increase in temperature and reached a maximum at 40°C and decreased thereafter (Figure 9). Removal was complete within 6 min at 40°C , while at higher temperatures it was incomplete possibly due to inactivation of protease. No hydrolysis was observed at lower temperatures. Gelatin removal was observed over a wide pH range of 7 to 11 with maximum hydrolysis at pH 10 (Figure 10) Hydrolysis was complete within 6 min at pH 10. Degradation took longer time at pH 7-10, or at 30 and 50°C . Complete hydrolysis was achieved by *Bacillus firmus* protease at 40°C , where less heating energy would be required. As gelatin hydrolysis was optimum at 40°C and pH 10, all further the experiments were carried out under these conditions.

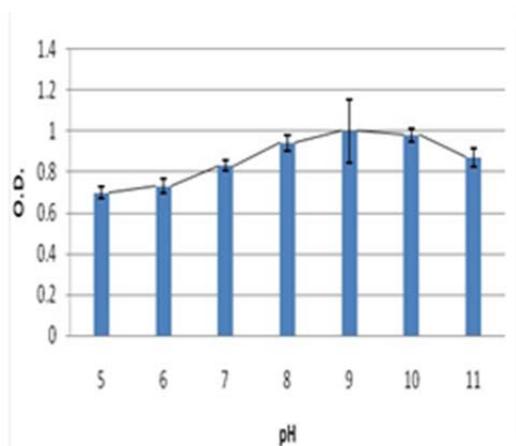


Figure 9: Effect of pH.

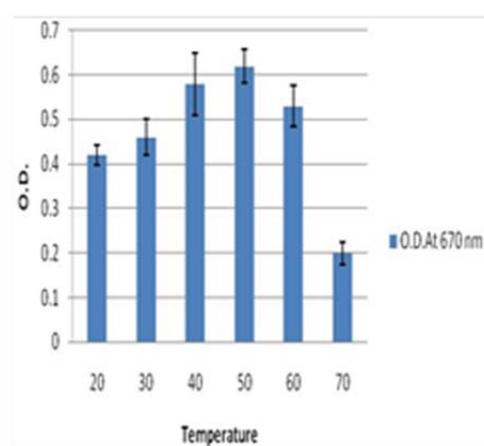


Figure 10: Effect of temperature.

Effect of enzyme concentration: Higher the enzyme concentration, greater was the hydrolysis rate (Clarke & Kirner, 1941). Effect of protease concentration on hydrolysis of gelatin was studied at 40°C and pH 10. The gelatin layer was completely stripped within 10 min. Longer incubations were required for complete removal of gelatin with lower enzyme concentrations. Complete gelatin removal occurred only after 35 min with the lowest enzyme concentration. From Figure 11, we can say that Hydrolysis percentages were 71.1 and 80.2 for 1.35 and 2.7 Uml^{-1} respectively at the end of 5 min.

Time course of hydrolysis and release of protein and hydroxyproline: In 6-7 min at pH 10 and 40°C 80-90% of stripping off of gelatin from x-ray film was achieved. With progress of gelatin removal protein and hydroxyproline in the hydrolysate increased. The ratio of hydroxyproline to protein was nearly constant. After complete stripping off of gelatin, hydroxyproline and protein concentrations ranged between $40\text{-}45 \mu\text{g ml}^{-1}$ and $160\text{-}170 \mu\text{g mg}^{-1}$ respectively (Figure 12).

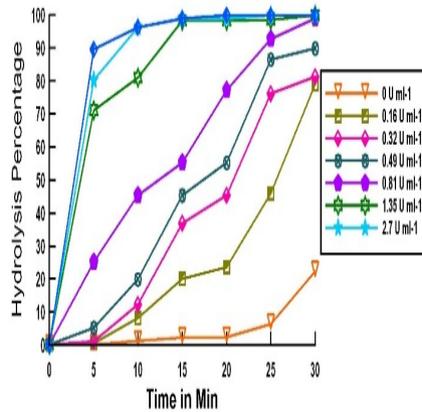


Figure 11: Effect of enzyme concentration.

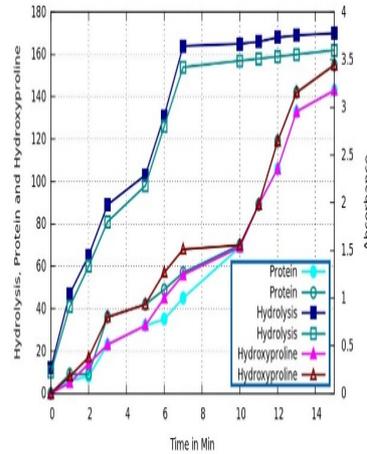
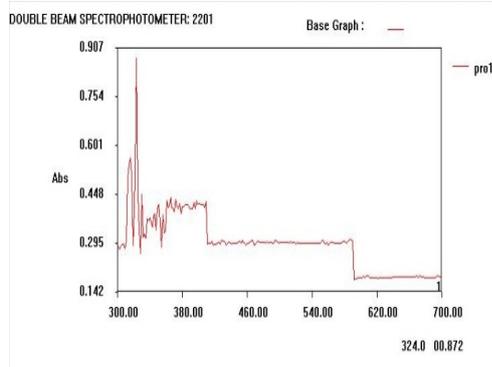
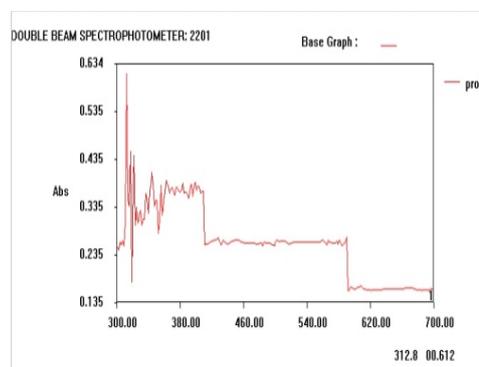


Figure 12: Time course of gelatin hydrolysis

Determination of silver nano-particles: The activity of Silver nanoparticles (Kulthong *et al.*, 2010) was determined by using UV-Double Beam Spectrophotometer (UV-1601, UV visible spectrophotometer, Shimadzu.). The maximum absorbance was shown by Samples 1 and 2, i.e., 0.872 and 0.612 respectively at 660 nm (Figure 13).



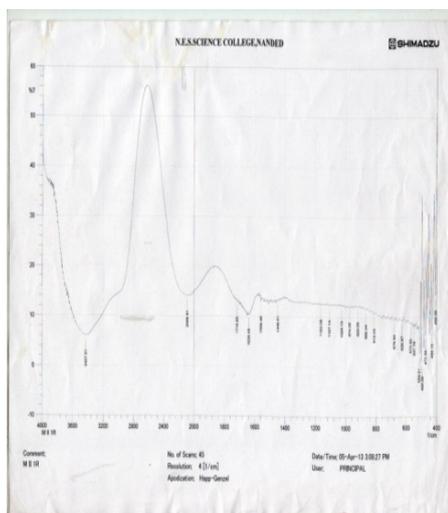
(a) Sample 1



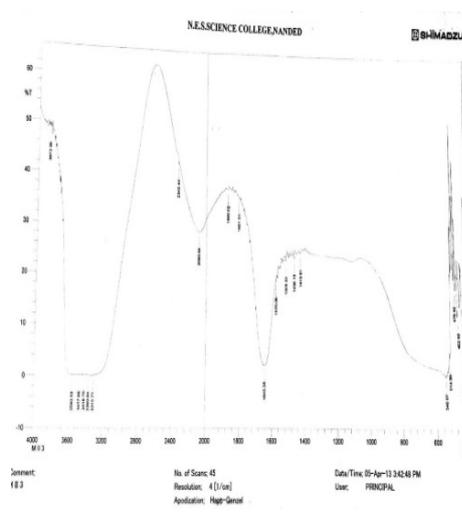
(b) Sample 2

Figure 13: UV-Visible absorption Spectra of silver nanoparticles.

Determination of alkaline protease: The presence of proteins on nanoparticle surface was also confirmed by FTIR (Fuerstenau *et al.*, 1983) (Figure 14). The band around 2,400 and 2,600 cm^{-1} is due to C-N (amide II) and C=N (amide I) bond respectively which arise due to the carbonyl stretch and -N-H stretch vibrations respectively in the amide linkages of the proteins. This indicates the presence of capped protein in nanoparticles.



(a) Sample 1



(b) Sample 2

Figure 14: FTIR spectrum of silver nano particles.

4. CONCLUSION

A critical need in the field of nanotechnology is the development of reliable and eco-friendly processes for synthesis of metallic nanoparticles. Here, we have reported a simple biological and low-cost approach for preparation of stable silver nanoparticles. In the present study attempt was made to investigate the ability of *Bacillus firmus* for the synthesis of silver nanoparticles by using X-ray films. The effect of pH, temperature & enzyme concentration was studied on hydrolysis of X-ray film along with enzyme solution. The silver content in the hydrolysate was determined by atomic spectra. Silver from the hydrolysate was recovered either as metallic silver or as silver chloride

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AN INVESTIGATION OF USING PALM BIODIESEL BLEND ON THE PERFORMANCE OF A STATIONARY MARINE DIESEL ENGINE

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ABSTRACT

Most of marine vehicles use petroleum based fuel as the source of energy in their daily operations. However, volatile oil price and concerns over depletion of petroleum resources have hastened researchers to look for other alternative fuel. As a result, biodiesel has been identified as one of the environmentally friendly alternatives energies and at the same time can reduce our dependency on petroleum-based fuels. The present study investigates the effects of palm biodiesel blends on stationary marine diesel engines with regards to the engines' performance characteristics, such as brake power, brake specific fuel consumption, exhaust gas temperature, brake thermal efficient and NO_x emissions. The experiment was conducted using Cummins NT-855 marine diesel engines. The results revealed that the use of palm biodiesel blends increased the brake specific fuel consumption, NO_x emissions and exhaust gas temperature up to 18.75%, 26.15% and 5.5% respectively. On the other hand, it contributed to reduced brake thermal efficiency by 12.17%. On one hand, palm biodiesel does not give any significant difference in engine brake power when compared to petroleum diesel. On the whole, it is proved that low blend palm biodiesel can be used in marine diesel engines, thereby providing a viable alternative to petroleum diesel.

Keywords: *Palm biodiesel; marine diesel engine; engine performance; emission.*

1. INTRODUCTION

Most modern ships use marine diesel engine as their prime mover due to their operating simplicity, robustness and fuel economy as compared to most other propulsion mechanisms. In fact, marine engines are very similar to the self-ignition engines in heavy-duty vehicles but they are generally larger, more complex, and operate with higher efficiency (Palocz-Andresen, 2013). Marine engines provide major power sources for sea transportation and contribute to the prosperity of the worldwide economy. The most types of marine fuel are marine diesel oil (MDO) and heavy fuel oil (HFO), which are derived from petroleum distillation. However, the volatility of crude oil prices lately and concerns over the depletion of fossil fuel reserves have forced researchers to look for other alternative fuels (Ashraful *et al.*, 2014; López *et al.*, 2015). As a result, biodiesel has gained a growing interest as one of the most promising solutions for these issues. Biodiesel is considered as a better option as it is renewable and eco-friendly as compared to fossil fuels. Its primary advantages are that it is biodegradable, renewable, sulphur-free and does not produce hazardous toxic gases (Demirbas, 2009). Biodiesel is briefly defined as the monoalkyl esters of vegetable oils or animal fats. It can be produced from a great variety of feedstocks, which include most common vegetable oils (e.g., soybean, cottonseed, palm, peanut, rapeseed, canola, sunflower, safflower, coconut) and animal fats as well as waste oils (Knothe *et al.*, 2005). The process used to convert this feedstock to biodiesel is called *transesterification* where the vegetable oil or animal fat is subjected to the chemical reaction. Fuel properties and combustion characteristics of biodiesel are similar to those of petroleum-derived diesel, which means that biodiesel can be used as an alternative fuel in any proportion without requiring any

engine modification (Demirbas, 2005, 2009; Shojaeefard *et al.*, 2012; Cirak & Demirtas, 2014; Chong *et al.*, 2015).

In recent years, biodiesel from palm oil and jatropha has been identified as renewable energy source with huge potential in the future (Mekhilef *et al.*, 2011). Palm biodiesel has been chosen for the current study because Malaysia is one of the world's leading palm oil producers. Most of its agricultural land is used for palm oil plantations. Oil palm planted area in 2015 reached 5.64 million ha, an increase of 4.6% as against 5.39 million ha recorded in the previous year (Malaysian Palm Oil Board, 2016). The palm oil harvested and produced from palm trees is referred to as crude palm oil (CPO). The oil is converted into biodiesel or palm methyl ester (PME) by the *transesterification* process using methanol as a catalyst. In order to encourage the use of palm oil biodiesel in industrial sectors in Malaysia, the National Biofuel Policy (NBP) was launched in August 2005 to promote the use of sustainable energy sources including biodiesel and biomass with support in terms of subsidized prices for the industry (Ministry of Plantation Industries and Commodities Malaysia, 2006). Since 2011, the B5 biodiesel (5% PME blend with 95% petroleum) was made available throughout the country and as for early 2016, B7 palm biodiesel (7% PME blend with 93% petroleum diesel) has been sold in the domestic market, especially for road vehicles (Chin, 2011).

Numerous studies on the application of biodiesel on diesel engines have been carried out and the results have shown that the performance of engines is comparable to that of using petroleum diesel fuel (Xue *et al.*, 2011; Roy *et al.*, 2014; Rizwanul Fattah *et al.* 2014; Wan Nor Maawa *et al.*, 2015; Rakopoulos *et al.*, 2015; Iqbal *et al.*, 2015; Senthilkumar *et al.*, 2015; Yasin *et al.*, 2015; Ali *et al.*, 2016; Monirul *et al.*, 2016; Rashed *et al.*, 2016;). These results are mostly obtained from laboratory experiments conducted on automotive or land-based diesel engines. The application of biodiesel on marine engines also has been explored by some researchers. A series of experiments was carried out by Murillo *et al.* (2007) on four-stroke, direct injection and naturally aspirated single cylinder outboard marine engines. They investigated the effect of different proportions of biodiesel that was derived from waste cooking oil to measure engine performance and emissions. The test fuels involved were pure diesel (B0), 10% biodiesel, 90% diesel (B10), 30% biodiesel, 70% diesel (B30), 50% biodiesel, 50% diesel (B50) and pure diesel (B100). The authors reported the test biodiesel reduces the brake power, in the extreme case of pure biodiesel (B100) up to 7.14% of the rated power. They claimed that waste cooking oil biodiesel also improved the emission of CO which up to 12%. On the other hand, there were increases in specific fuel consumption (about 11.4%) and brake thermal efficiency. The author addressed that the reason may be related to the atomisation of the blend during injection or with the stability of the mixtures of fuels during storage, pumping and injection.

Another study using the same biodiesel feedstock was conducted by Roskilly *et al.* (2008) on marine engine on-board ships. The tests were performed on a Perkins 404C-22 engine in Ship No. 1 and a Nanni Diesel engine 3.100HE in Ship No. 2. The result showed that biodiesel increased brake specific fuel consumption (BSFC) by 8.6-20.9% for both marine engines. The author identified this as mainly due to the lower gross heating values of biodiesel (39.62– 39.66 MJ/kg) compared with that of petroleum diesel (45.00 MJ/kg). The exhaust temperatures were a little higher when fuelled with biodiesel, ranging from 1.8% to 11.5% for both engines. Engine output powers for both tested engines were nearly equal with the difference of less than 1% on the whole test range. However, interestingly the author found that biodiesel reduced the NO_x emission up to 24.3%. These results contradicted with Murillo *et al.* (2007). They suggested that the reason for this reduction is due to smaller heating values and the higher Cetane number of biodiesel. Early studies on a Wärtsilä marine diesel engine using palm biodiesel was reported by Juoperi (2008). The results indicated a slight increase in NO_x, and a reduction of CO and HC exhaust emissions. More recently, Lin (2014) examined the effects of blending fishing boat fuels with various weight proportions of waste cooking oil biodiesel. The results showed that biodiesel blending can significantly improve the inferior fuel properties such as increasing in flash point and a reduction in sulphur content. However, the author reported that biodiesel blending caused a slight decrease in heating value around 1–4.5%.

It should be noted from the above literature that there are limited researches on marine diesel engines with palm biodiesel have been reported so far, and this has motivated the present study. Furthermore, the existence of the Malaysian national biofuel policy has increased the exploration of biodiesel research related to resources available in the country. Therefore, it is a great interest for the authors to investigate the effects of palm biodiesel on the marine diesel engine with regards to the engines' performance and emission characteristics. The authors hope that this paper can provide useful information to researchers, engineers, marine industry players and those interested in biodiesel as an alternative energy source rather than rely solely on fossil fuels.

2. EXPERIMENTAL SETUP

In this study, the experiments were performed on a stationary Cummins NT-855 marine diesel engine. The details of engine specification are presented in Table 1. Full setup of marine diesel engine is shown in Figure 1, where 250 kW eddy-current dynamometer was attached to the engine in order to measure engine brake power and torque. Fuel consumption and air flow rate was measured by positive displacement type KOBOLD flowmeter and TAYLOR air flowmeter respectively. The engine was equipped with K-type thermocouples and resistance temperature detectors (RTD) with cover temperature range from $-40\text{ }^{\circ}\text{C}$ to $1200\text{ }^{\circ}\text{C}$ for temperature measurement. All the required data were collected through REO-DCA data acquisition unit as shown in Figure 2. The whole schematically diagram of experimental setup is given in Figure 3

Table 1: Marine diesel engine specifications.

Brand/Model	Cummins NT-855
Engine type	4 cycle-Inline-6 cylinder
Bore x stroke	139 mm x 152 mm
Displacement volume	14 liter
Compression ratio	14.5
Maximum torque	1068 N.m
Maximum power	201 kW
Cooling system	Water cooling

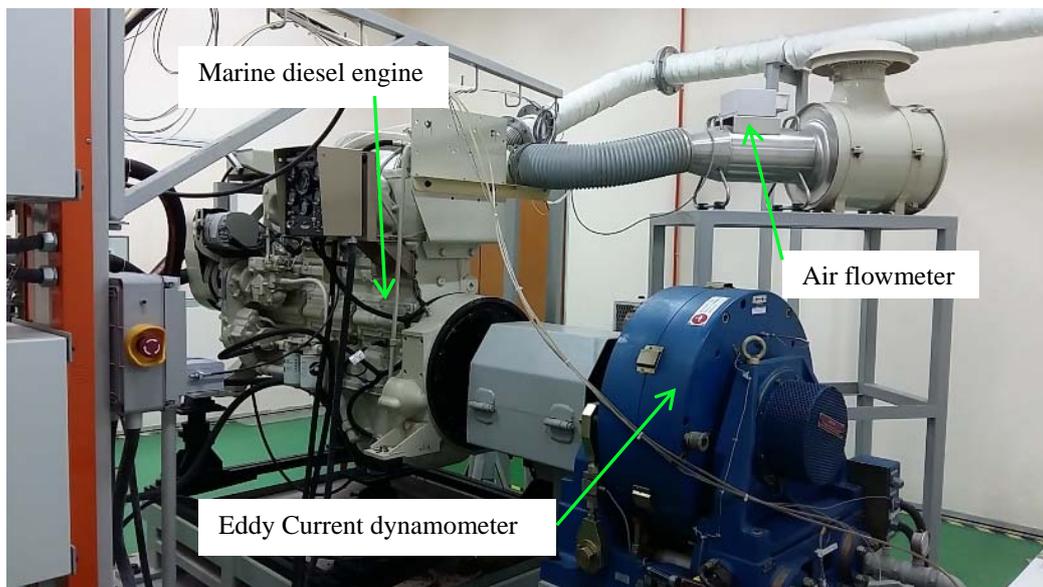


Figure 1: Full setup of marine diesel engine.



Figure 2: REO-DCA data acquisition unit.

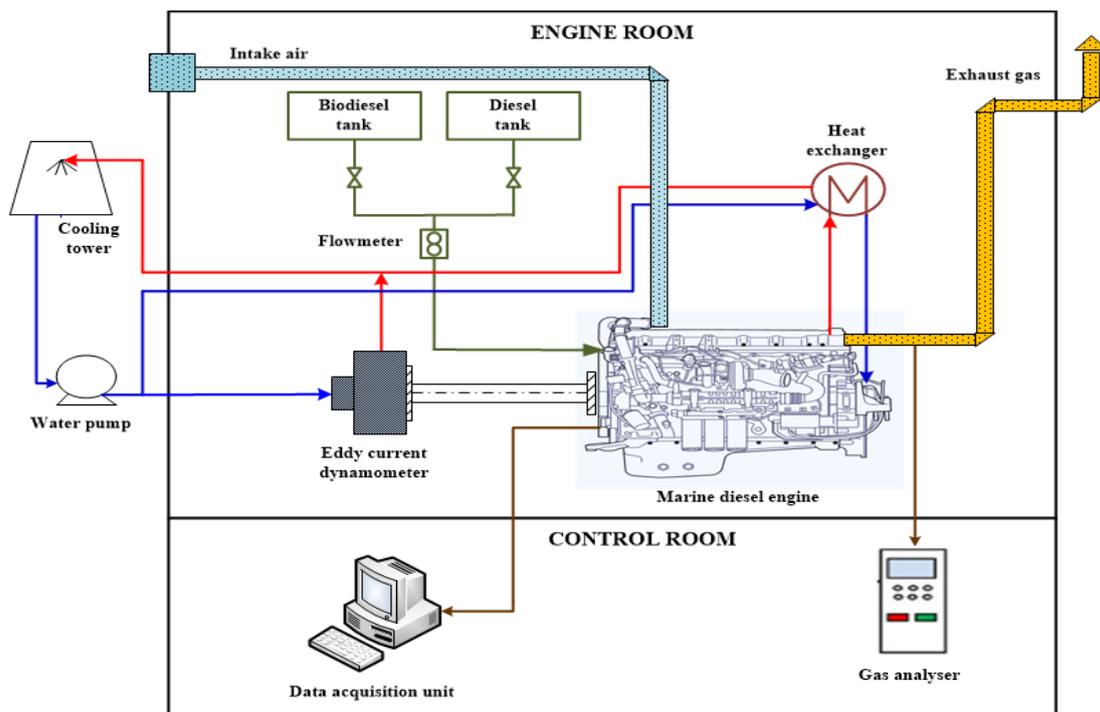


Figure 3: Schematic diagram of the experimental setup.

The experiments were carried out using low percentage of palm biodiesel blend such as B0 (100% petroleum diesel), B5 (5% biodiesel + 95% petroleum diesel), B10 and B15. The blend samples were prepared by mixing petroleum diesel with certified palm oil biodiesel. The blended fuel samples are shown in Figure 4. Basic properties of all tested fuels were measured and summarised in Table 2. Engine performance tests were performed under steady-state condition at three different engine loads (10%, 30% and 50%) and constant speeds of 1600 rpm. Before each test, the engine was warmed up for about 15 minutes until the cooling water temperature stabilized. Desired parameters such as engine speeds, torque, brake power, exhaust gas temperature and fuel consumption were recorded, while brake specific fuel consumption, brake thermal and volumetric efficiency were computed later. All tests were completed without any modifications on the test engine.



Figure 4: Biodiesel blend fuel samples.

Table 2: Basic properties of palm biodiesel fuel blends.

Fuel properties	B0	B5	B10	B15	B100
Heating value (MJ/kg)	48.26	46.87	46.33	45.47	42.07
Density @ 15 °C (kg/m ³)	813	815	818	820	854
Kinematic viscosity @ 40 °C (cst)	4.02	4.15	4.52	4.60	5.66
Flash point (°C)	70	104	110	113	180
Cetane number	46	47	48	49	57

3. RESULTS AND DISCUSSION

3.1 Brake Power

Brake power is the measurement of an engine's horsepower before the loss in power caused by the gearbox, alternator, water pump, and other auxiliary components. Brakes refer to a device used to load an engine and hold it at a desired engine speed. During testing, the output torque and rotational speed can be measured to determine the brake horsepower which is the actual shaft horsepower and is measured by using dynamometer. Figure 5 illustrates the brake power of the marine diesel engine running with palm biodiesel blends with respect to three engine loads (10%, 30% and 50%) and at constant engine speed of 1600 rpm. Since the amount of injected liquid fuel increased with engine load, the engine power rose by increasing engine load for all tested fuels. The brake power at 10% engine load for B0, B5, B10, and B15 were 17.0 kW, 18.0 kW, 17.4 kW and 17.7 kW respectively. These values were increased to 83.9 kW, 83.9 kW, 83.8 kW and 82.9 kW at half load (50%) condition. Meanwhile, the results show that there are no significant differences in the measured engine power output between petroleum diesel and biodiesel blends for each load tested except some drop for B10 fuel at 30% engine load. According to (Xue et al. 2011) about 22% of previous finding stated that biodiesel produced similar power output compared to diesel. In this study, all tested palm biodiesel blends produced almost the same output power compare to diesel, thus this indicate that palm biodiesel blend can be used for marine diesel engines without scarifies any reduction of engine brake power.

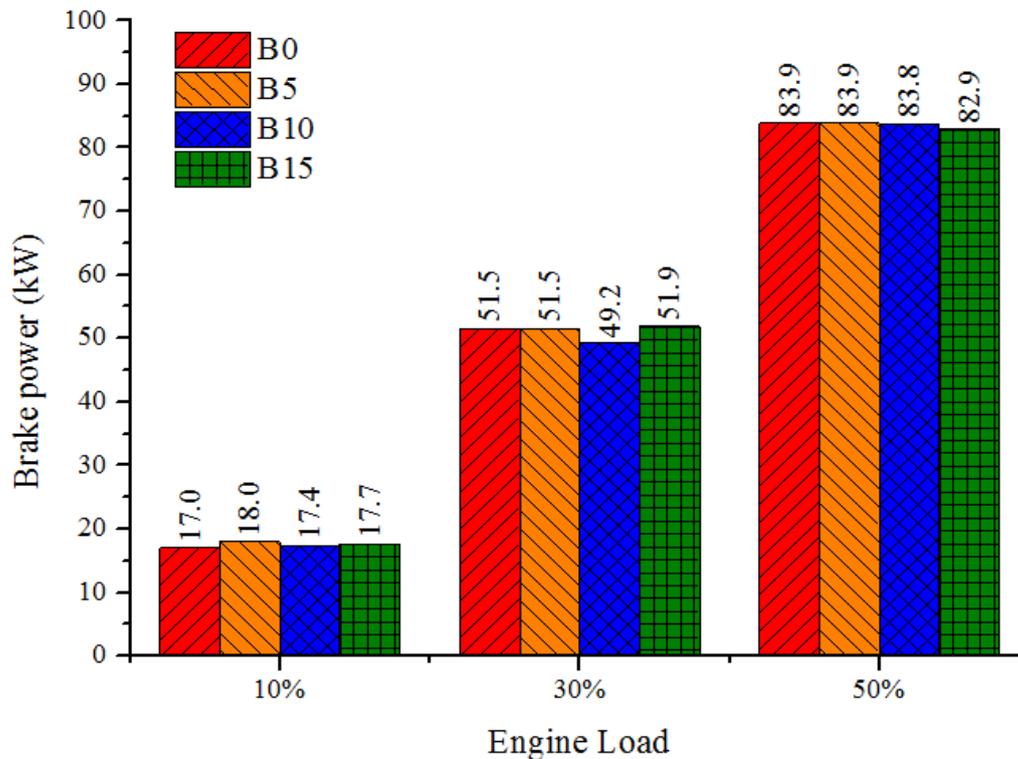


Figure 5: Brake power at various engine loads.

3.2 Brake Specific Fuel Consumption

Brake specific fuel consumption (BSFC) is defined as the fuel consumption rate divided by its corresponding engine brake power output. It is desirable to obtain a lower value of brake specific fuel consumption meaning that the engine used less fuel to produce the same amount of work. The variations of BSFC with engine load for B0, B5, B10 and B15 are shown in Figure 6. BSFC, in general, was found to decrease with an increasing of engine loads in all blends. One possible reason for this trend could be where increase percentage of required fuel in engine operation is less than the increase percentage of brake power due to relatively less portion of the heat losses at higher load. On the other hand, the increase proportion of biodiesel in tested fuel has raised BSFC value which means more amount of fuel required for the same operation. Maximum raised of fuel consumption which is about 18.75% was noticed at 10% engine load when compared to petroleum diesel. The blend with higher biodiesel content consumed more by the engine as it needed to compensate the loss of heating value in biodiesel (Shojaeefard et al. 2012). Therefore, the amount of fuel introduced to the cylinder for a desired energy input has to be greater with the biodiesel blends. Furthermore, higher containments of oxygen in biodiesel are also the cause of the lower heating value. Despite the better combustion of biodiesel compared to the petroleum diesel, the oxygen in biodiesel takes up space in the blend and slightly increases the fuel consumption rate. The increasing of BSFC with palm biodiesel mixture in diesel engine was consistent with findings of past studies by (Monirul et al. 2016; Rizwanul Fattah et al. 2014; Rashed et al. 2016; Ali et al. 2016; Murillo et al. 2007; Roskilly et al. 2008).

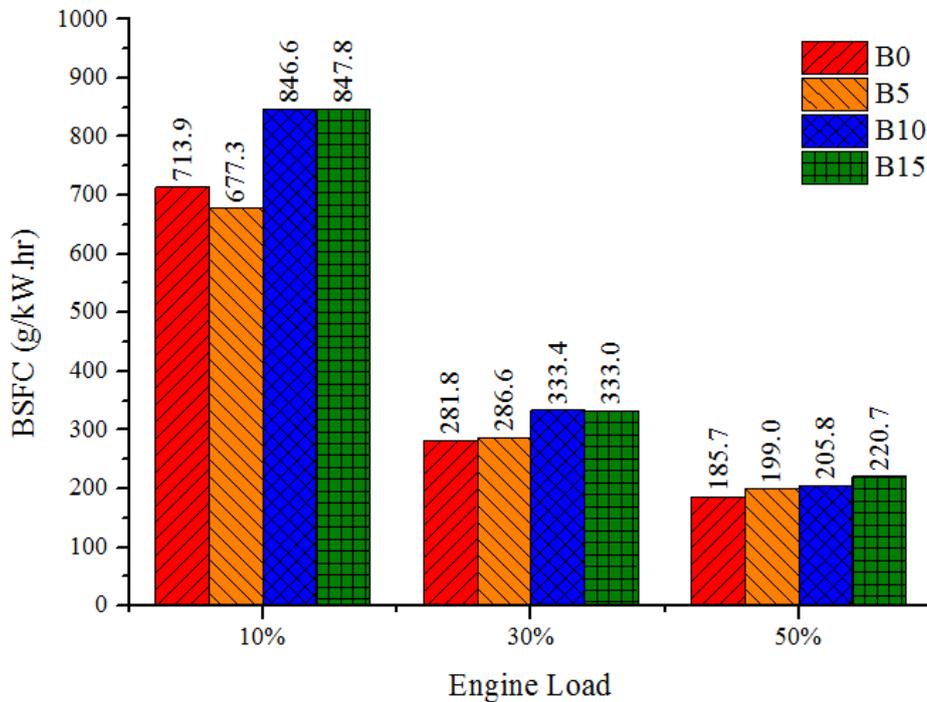


Figure 6: Brake specific fuel consumption various engine loads.

3.3 Exhaust Gas Temperature

The variations of exhaust gas temperature (EGT) of marine diesel engine, when operated with diesel-biodiesel blends at three different engine loads, are shown in Figure 7. EGT was found to be increase with the increase in both concentrations of palm biodiesel in the blends and engine load. At 10% engine load conditions, EGT was 158.9 °C, 160.6 °C, 160.3 °C and 167.5 °C with B0, B5, B10 and B15 respectively, whereas it was increased to 277.6 °C, 278.4 °C, 278.1 °C and 280.4 °C at 50% engine loading. The increase in EGT with engine load is obvious from the simple fact that the higher amount of fuel was required in the combustion chamber in order to generate extra power needed to take up additional loading. Palm biodiesel blend has slightly contribute to the increasing of EGT when compare to petroleum diesel which is observed less than 5.5% increment for all engine load conditions. This phenomenon is due to high oxygen content in palm biodiesel which contribute to more amounts of heat to be reclaimed during combustion process. Most of biodiesel will slightly increase in EGT (Kumar & Chauhan 2013).

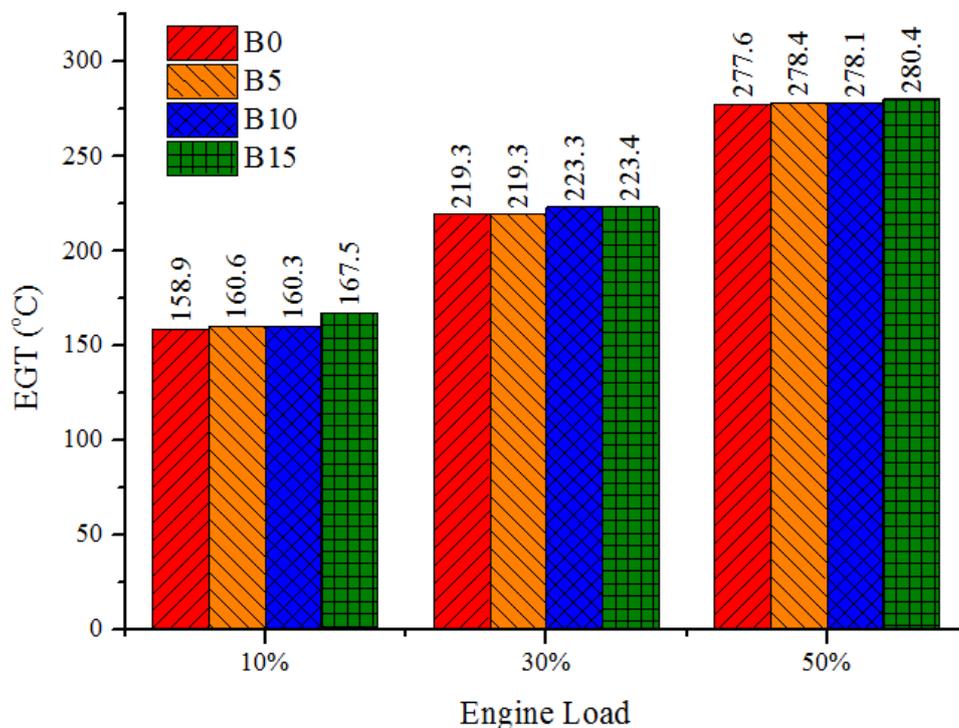


Figure 7: Exhaust gas temperature at various engine loads.

3.4 Brake thermal efficiency

Brake thermal efficiency (BTE) is used to evaluate how well an engine converts heat from supplied fuel to mechanical energy. This parameter is determined by dividing the brake power of the engine to the amount of energy input to the system. The amount of energy which cannot be converted into mechanical energy is discharged by the system through friction losses, heat transfer through the engine cylinder and exhaust gases. BTE of marine diesel engine, when operated with diesel-biodiesel blends at different engine loads, has been plotted in Figure 8. At 10% engine loading, brake thermal efficiency values were found to be 10.4%, 11.3%, 9.2% and 9.3% with B0, B5, B10, and B15, respectively, which were increased to 39.8%, 38.6%, 37.8% and 35.9% at half load (50%) condition. The BTE improved with the increasing engine load for the main reason that a relatively less portion of the power was lost at higher loads. The figure also indicates that BTE reduced when increase palm biodiesel percentage in blended fuel, which is up to 12.17% reduction. The decreasing of BTE is due to higher density and viscosity value of palm biodiesel compared to petroleum diesel (Öztürk 2015). This will reduced the atomization and vaporization of biodiesel blends fuel which producing uneven combustion characteristics compared with diesel (Banapurmath et al. 2008).

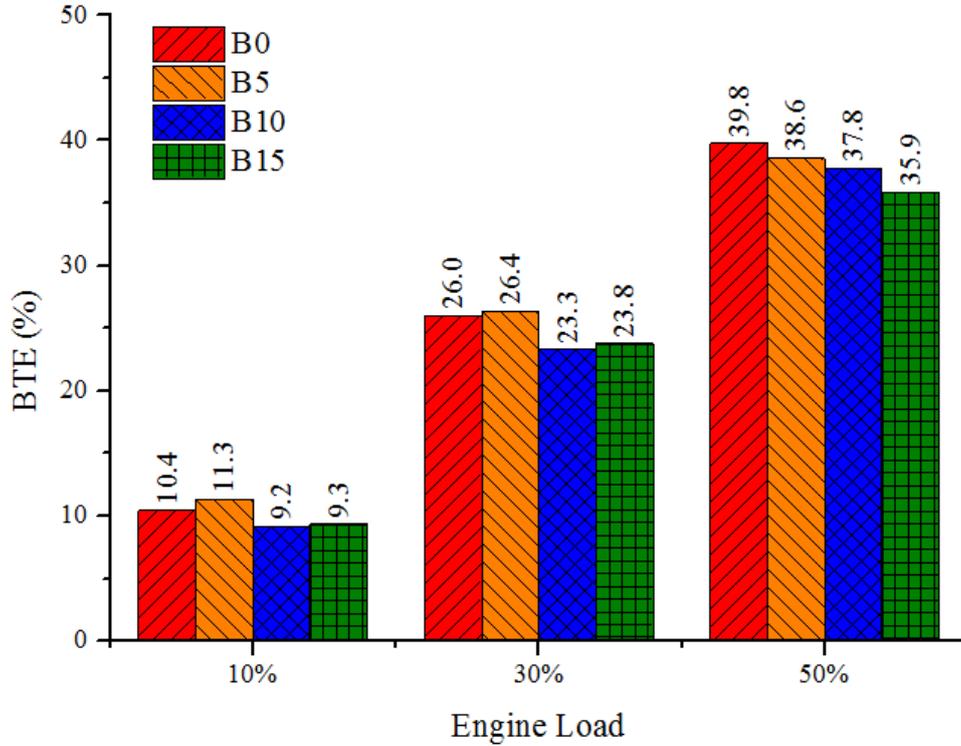


Figure 8: Brake thermal efficiency at various engine loads.

3.5 Nitrogen Oxide Emissions (NO_x)

NO_x emissions are resulted from combustion process when nitrogen and oxygen are present at elevated temperatures in engine cylinder (Can 2014). Kinetics of NO_x formation is governed by Zeldovich mechanism and its formation is highly dependent on temperature and availability of oxygen (Heywood 1988). Figure 9 shows the effect of biodiesel blend on NO_x emission at three different engine loads. It is found that NO_x emissions increased up to 26.15% when more percentage of palm biodiesel added in blends. This result correlates with a slightly higher EGT with biodiesel. Biodiesels contain higher oxygen component (Roskilly et al. 2008; Öztürk 2015; Palash et al. 2013), thus it is evident that there is higher oxygen content will react with the nitrogen component from intake air, resulting in extra NO_x formation. Fattah et al. state that NO_x will increases with palm methyl ester content in blend (Rizwanul Fattah *et al.*, 2013).

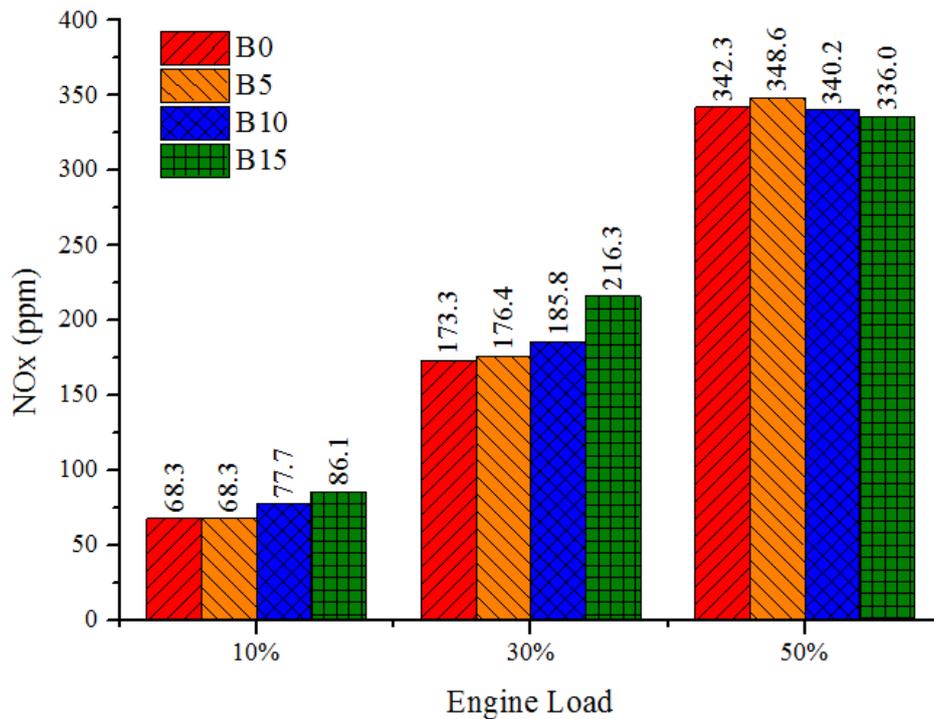


Figure 9: Nitrogen oxide emissions at various engine loads.

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MORPHOMETRIC ANALYSIS OF THE MAZANDRAN RIVER WATERSHED, IRAN

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ABSTRACT

In this study, the Mazandran River watershed is selected for detailed morphometric analysis. Morphometric analysis for parameters of stream number (N_u), stream order (U), cumulative length of streams (L), bifurcation ratio (R_b), watershed relief (B_b), drainage density (D_d), stream frequency (F_s), form factor (R_f), circularity ratio (R_c) and elongation ratio (R_e) is carried out to describe the drainage properties of the three subwatersheds in the study area (W1, W2 and W3). The results show that among the subwatersheds, the highest N_u is 333 (stream order 1 for W1), while the lowest N_u is 1 (stream order 4 for W1). For U , among the subwatersheds, W1 and W2 are of order 3, and W3 is of order 4, showing that they are in the early stages of erodibility. W1 has the highest L of streams (5.72 km), whereas W3 has the lowest L of streams (3.11 km). The R_b values for the subwatersheds are between 0.73 to 1.84, while D_d varies between 0.0055 and 0.0060 km/km². The results also show that the F_s values for the subwatersheds exhibit positive correlations with D_d (from 0.48 to 0.58). The minimum and maximum form factors (R_f) are observed for W3 (0.58) and W2 (0.93) respectively, indicating W2 is more round, resulting in lower lag time. R_c is in the range of 0.43 to 0.75. The overall results indicate that in the subwatersheds are have high erodibility and need more management.

Keywords: *Morphometric analysis; subwatersheds; drainage properties; Strahler's ordering method; erodibility.*

1. INTRODUCTION

Morphometric analysis of watersheds and drainage networks plays a vital role in understanding geo-hydrological behavior and expressing the prevailing climate, geomorphology and structural antecedents of terrains. It involves the evaluation of streams through the measurement and analysis of various stream and drainage parameters to predict the approximate behavior of the watersheds during periods of heavy rainfall (Verstappen, 1983; Kumar *et al.*, 2000; Parveen *et al.*, 2012).

Remote sensing and geographical information systems (GIS) have proven to be effective tools for morphometric analysis (Rao *et al.*, 2010). Digital elevation models (DEMs) have provided a precise, fast, and inexpensive way for analyzing hydrological systems in GIS-based evaluation (Smith & Sandwell, 2003). The processed DEMs are used for extracting stream networks and other layers (Mesa 2006; Magesh *et al.*, 2011, Mokarram & Sathyamoorthy, 2015), which are then used to deduce morphometric parameters such as drainage watershed area, drainage density, drainage order, relief and network diameter (Kumar *et al.*, 2014).

The present study aims at using remote sensing and GIS technologies to compute various parameters of morphometric characteristics of the Mazandran River watershed. The study of stream morphometric properties of the study area is important to determine effective strategies for storage of water and preventing floods (Darvishan *et al.*, 2008).

2. METHODOLOGY

2.1 Study Area

The study area consists of three subwatersheds (W1 to W3), which are shown in Figure 1. The data used for the study is a Shuttle Ray Topographic Mapping (SRTM) DEM with resolution of 30 m. The altitude of the study area ranges from its lowest of 1,407 m to the highest of 3,591 m with area of 2,288.27 km².

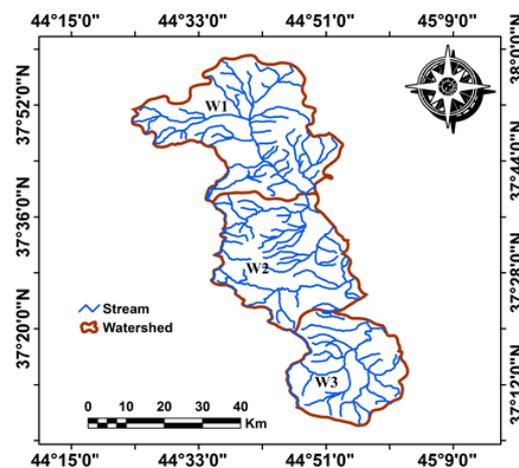


Figure 1: Location map of the study area.

2.2 Extraction of Drainage Networks

Extraction of drainage networks from the DEM is done using the flow direction method, which consists of the following steps (O'Callaghan & Mark, 1984):

- i. *Fill Sinks*: A sink is an uncompleted value lower than the values of its neighborhood. To ensure proper drainage mapping, these sinks are filled by increasing elevations of sink points to their lowest outflow point.
- ii. *Calculate Flow Direction*: Using the filled DEM produced in the Step i, the flow directions are calculated using the eight-direction flow model, which assigns flow from each grid cell to one of its eight adjacent cells in the direction with the steepest downward slope.
- iii. *Calculate Flow Accumulation*: Using the output flow direction raster created in Step ii, the number of upslope cells flowing to a location is computed.
- iv. *Define Stream Network*: The next step is to determine a critical support area that defines the minimum drainage area that is required to initiate a channel using a threshold value.
- v. *Stream Segmentation*: After the extraction of drainage networks, a unique value is given for each section of the network associated with a flow direction.

2.3 Morphometric Analysis

Strahler's system of stream analysis (Strahler, 1964) is probably the simplest and most used system, and hence, is used for this study. According to this method, each finger-tip drainage network is designated as a segment of the first order. At the connection of any two first-order segments, a network of the second order is produced, which extends down to the point where it joins another second order river, where upon a segment of the third order results.

The extracted drainage networks are ordered using Strahler's ordering method and transformed to vector layer for further analysis. The morphometric parameters that are analyzed in this study are shown in Table 1. According to Table 1, morphometric analysis is conducted for parameters of stream order (U), stream length (L_u), mean stream length (L_{sm}), stream length ratio (R_L), bifurcation ratio (R_b), mean bifurcation ratio (R_{bm}), relief ratio (R_h), drainage density (D_d), stream frequency (F_s), drainage texture (R_t), form factor (R_f), circularity ratio (R_c), elongation ratio (R_e) and length of overland flow (L_g). The analysis is carried out to describe the drainage properties of the three subwatersheds in the study area.

Table 1: Morphometric parameters used for this study.

No.	Morphometric parameters	Formula	Description	Reference
1	Stream order (U)	Hierarchical rank		Strahler (1964)
2	Stream length (L_u)	Length of the stream		Horton (1945)
3	Mean stream length (L_{sm})	$L_{sm} = L_u / N_u$	N_u = Total number of stream segments of order u	Strahler (1964)
4	Stream length ratio (R_L)	$R_L = L_u / L_{(u-1)}$	$L_{(u-1)}$ = Total stream length of the next lower order	Horton (1945)
5	Bifurcation ratio (R_b)	$R_b = N_u / N_{(u+1)}$	$N_{(u+1)}$ = Number of segments of next higher order	Schumms (1956)
6	Mean bifurcation ratio (R_{bm})	R_{bm} = Average R_b of all orders		Strahler (1957)
7	Relief ratio (R_h)	$R_h = H / L$	H = Total relief (relative relief) of the watershed in km; L_b = Watershed length	Schumms (1956)
8	Drainage density (D_d)	$D = L_u / A$	A = Watershed area (km^2)	Horton (1932)
9	Stream frequency (F_s)	$F_s = N_u / A$		Horton (1932)
10	Drainage texture (R_t)	$R_t = N_u / P$	P = Watershed perimeter (km)	Horton (1945)
11	Form factor (R_f)	$R_f = A / L_b^2$		Horton (1932)
12	Circularity ratio (R_c)	$R_c = 4\pi * A / P^2$		Miller (1953)
13	Elongation ratio (R_e)	$R_e = (2 / L_b) * (A / \pi)^{0.5}$	π = Pi	Schumms (1956)
14	Length of overland flow (L_g)	$L_g = 1 / D * 2$		Horton (1945)

3. RESULTS & DISCUSSION

The morphometric parameters for the study area are calculated and the results are given in the Table 2. Automatic extraction of streams is conducted using the SRTM DEM (Figure 2). The SRTM DEM is also used to prepare slope, aspect and contour maps. Based on the stream order, the Mazandran River watershed is classified as a three-order watershed to interpret the morphodynamic parameters as shown in Table 1 (Horton, 1932, 1945; Smith, 1950; Strahler, 1964; Sreedevi *et al.*, 2005; Mesa 2006).

The ranking of streams in the study area is done based on the Strahler method. The stream orders are classified up to 4th orders (Figure 3a). Further information on the stream orders of the subwatersheds are shown in the Table 2.

Aspect refers to the direction a mountain slope faces (Kumar *et al.*, 2014). The value of the output raster data set shows the compass direction of the aspect (Magesh *et al.*, 2011). The aspect map of the watershed is shown in Figure 3a. As shown in Figure 3b, west facing areas have lower moisture content and high evaporation rate, while parts falling towards east-facing slopes have higher moisture content and lower evaporation rate (Kumar *et al.*, 2014).

Slope analysis is an important factor in geomorphological researches for watershed development and important for morphometric analysis (Magesh *et al.*, 2011; Gayen *et al.*, 2013; Kumar *et al.*, 2014). A slope map of the study area was calculated based on the SRTM DEM data using the spatial analysis tool in ArcGIS 10.3. The slope values ranges from 0 to 76.97 ° (Figure 3c). Higher slope degree results in rapid runoff and increased erosion rate with less ground water recharge potential. Higher slope is identified in the east and south parts of the study area.

Relative relief is an important morphometric parameter used to determine the morphological characteristics of any topography (Gayen *et al.*, 2013; Kumar *et al.*, 2014). The highest relative relief is calculated as 3,591 m, while the lowest value is recorded as 1,407 m, as shown in Figure 3d.

The count of stream channels in each order is termed as stream order (Horton, 1945). During computation, it is identified that the number of streams gradually decreases as the stream order increases; the variation in stream order and size of tributary watersheds largely depends on physiographical, geomorphological and geological conditions of the region (Kumar *et al.*, 2014). A total of 1,187 stream lines were recognized in the whole watershed, out of which 66.13 % (785) is 1st order; 28.05 % (333), 2nd order; 5.72%, (68); 3rd order; and 0.084 % (1), 4th order (Table 3).

The stream length is a measure of the hydrological properties of the bedrock and drainage extent. Wherever the bedrock and formation are permeable, only a small number of relatively longer streams are formed in a well-drained watershed. On the other hand, a large number of streams of smaller length are developed where the bedrocks and formations are less permeable (Horton 1945; Sethupathi *et al.*, 2011). The results of stream length are shown in Table 4.

The stream length ratio between the streams of different orders of the Mazandran River watershed shows a change in each subwatershed (Table 5). This change might be attributed to variation in slope and topography, indicating the late youth stage of geomorphic development in the streams of the watershed (Vittala *et al.*, 2004).

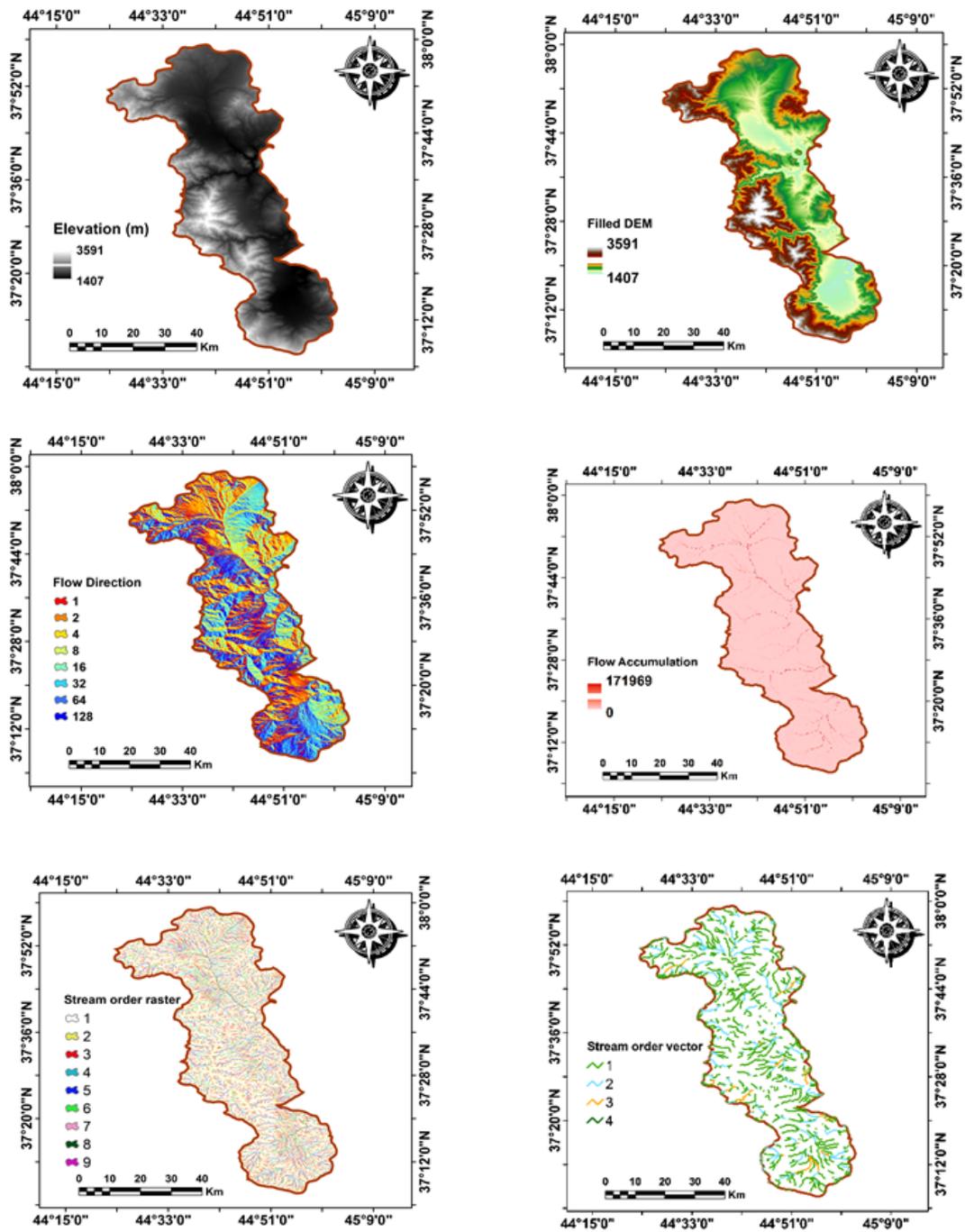


Figure 2: Automatic extraction of streams from the SRTM DEM.

Table 2: Areas of the subwatersheds of the study area.

Subwatershed	Stream order	Area (km ²)
1	III	948.57
2	III	781.75
3	VI	557.95

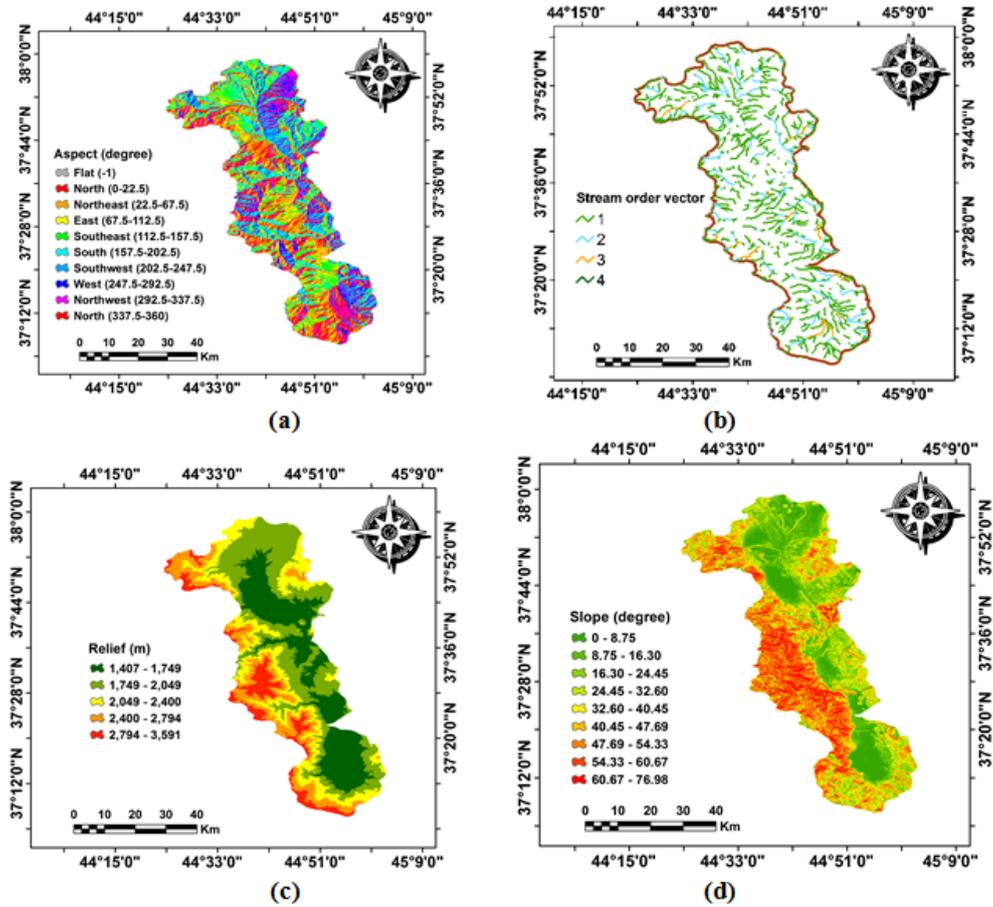


Figure 3: (a) Stream order, (b) aspect map, (c) slope map and (d) relief map of the study area.

Table 3: Stream numbers of the subwatersheds of the study area.

Subwatershed	Number			
	I	II	III	IV
1	333	134	22	-
2	247	102	24	-
3	205	97	22	1

Table 4: Stream length of the subwatersheds of the study area.

Subwatershed	Length (m)			
	I	II	III	IV
1	4.08	1.45	0.199	-
2	2.98	0.94	0.011	-
3	2.26	0.84	0.012	0.0065

Table 5: Stream length ratios of the subwatersheds of the study area.

Subwatershed	II/I	III/II	IV/III
1	0.90	0.83	-
2	0.77	1.20	-
3	0.79	1.38	0.54

According to Schumn (1956), the term bifurcation ratio (R_b) may be defined as the ratio of the number of the stream segments of given order to the number of segments of the next higher orders. In the watershed, the higher values of R_b indicate a strong structural control in the drainage pattern, whereas the lower values indicate that the subwatersheds are less affected by structural disturbances (Vittala *et al.*, 2004; Chopra *et al.*, 2005). The R_b values for the subwatersheds vary from 0.84 to 1.84 (Table 6).

Table 6: Bifurcation ratios of the subwatersheds of the study area.

Subwatershed	I/II	II/III	III/VI
1	1.11	1.20	-
2	1.30	0.84	-
3	1.26	0.73	1.84

The mean stream length (L_{sm}) values for the watershed range from 0.0065 to 0.012 km (Table 7). It is found that the L_{sm} value of any stream order is higher than that of the lower order and less than that of its next higher order in the watershed. Strahler (1964) showed that the L_{sm} is a characteristic property related to the size of drainage network and its associated surfaces. The mean stream lengths of the subwatershed based on stream order is shown in the Table 7.

Table 7: Mean stream lengths of the subwatersheds of the study area.

Subwatershed Stream order	Mean Stream Length (km)			
	I	II	III	IV
1	0.012	0.0108	0.0090	-
2	0.0120	0.0092	0.011	-
3	0.011	0.0087	0.0120	0.0065

Low values of relief ratio (R_h) are mainly caused by the persistent basement rocks of the watershed and low degree of slope (Mahadevaswamy *et al.*, 2011). R_h normally increases with decreasing drainage area and size of a given drainage watershed (Gottschalk 1964). The mean relief ratio of each subwatershed is shown in the Table 8.

Table 8: Relief ratios of the subwatersheds of the study area.

Subwatershed	Min Elevation	Max Elevation	Relief	Relief Ratio
1	1421	3360	1939	0.002955
2	1440	3591	2151	0.001828
3	1407	3453	2046	0.001524

Drainage density (D_d) is a measure the total stream length in a given watershed to the total area of the watershed (Strahler 1964). The drainage density is affected by the factors that control characteristic length of the watershed. Drainage density is related to various features of landscape dissection such as valley density, channel head source area, relief, climate and vegetation (Moglen *et al.*, 1998), soil and rock properties (Kelson & Wells, 1989), and landscape evolution processes. The drainage density values of the subwatersheds are shown in Figure 4 and Table 9. Low drainage density leads to coarse drainage texture, while high drainage density leads to fine drainage texture, high runoff and erosion potential of the watershed area (Strahler, 1964). Triangular irregular network (a), drainage density (b) and DTM from different angles for perspective views are shown in the Figure 4.

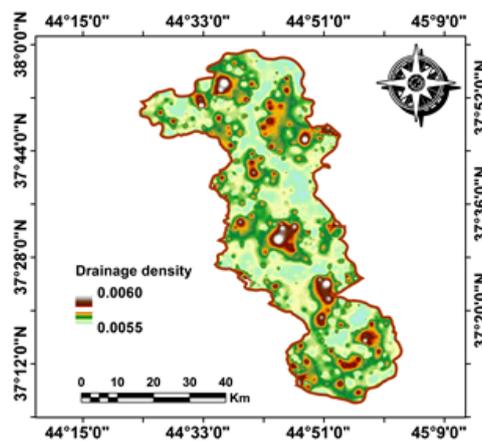


Figure 4: Drainage density map of the study area.

Table 9: Drainage densities of the subwatersheds of the study area.

Subwatershed	Total length	Area (km ²)	Drainage density
1	5.73	948.57	0.00604
2	3.93	781.75	0.00502
3	3.12	557.95	0.0055

Stream frequency (S_f) is the total number of stream segments of all orders per unit area (Horton, 1932). The stream frequency values of the Mazandran River watershed range from 0.48 to 0.52 (Table 10). Stream frequency mainly depends on the lithology of the watershed and reflects the texture of the drainage network.

Table 10: Stream frequencies of the subwatersheds of the study area.

Subwatershed	<i>N</i>	Area (km ²)	Stream frequency
1	489	948.57	0.52
2	373	781.75	0.48
3	325	557.95	0.58

Horton (1932) described form factor (R_f) as the ratio of the area of the watershed and square of the watershed length. The value of form factor would always be greater than 0.78 for perfectly circular watersheds. The smaller the value of form factor, the more elongated will be the watershed. The R_f values shown in Table 11 indicate that the Mazandran River watershed is elongated.

Table 11: Form factors of the subwatersheds of the study area.

Subwatershed	Length of watershed (km)	Area (km ²)	Form factor
1	37	948.57	0.69
2	29	781.75	0.93
3	31	557.95	0.58

Miller (1953) stated that circularity ratio (R_c) is the ratio of the area of the watersheds to the area of a circle having the same circumference as the perimeter of the watershed. Miller (1953) described R_c as an important ratio that shows the dendritic step of a watershed. The circularity ratio is mainly concerned with the length and frequency of streams, geological structures, climate, relief and slope of the watershed. As shown in Table 12, the R_c values of the subwatersheds range from 0.43 to 0.75.

Table 12: Circularity ratios of the subwatersheds of the study area.

Subwatershed	Perimeter (km)	Area (km ²)	Circularity ratio
1	166.69	948.57	0.43
2	130.44	781.75	0.58
3	96.73	557.95	0.75

Elongation ratio (R_e) is defined as the ratio of diameter of a circle having the same area as of the watershed and maximum watershed length (Schumm 1956). Higher values of elongation ratio show high infiltration capacity and low runoff, whereas lower R_e values are characterized by high susceptibility to erosion and sediment load (Reddy *et al.*, 2004). The values of R_e for the study area are shown in Table 13.

Table 13: Elongation ratios of the subwatersheds of the study area.

Subwatershed	Total length	Area (km ²)	Elongation ratio
1	5.73	948.57	0.94
2	3.93	781.75	1.09
3	3.12	557.95	0.86

The overall results of the morphometric analysis of the study area are shown in Table 14. The watershed has high relief ratio, indicating high elevation. The stream frequency is also high, showing that the watershed has high erodibility. Based on form factor and circularity ratio, it is determined that the watershed is stretched and has more lag time.

Table 14: Overall results of the morphometric analysis of the study area.

No.	Morphometric parameters	Stream order			
		I	II	III	IV
1	Stream order (U)	I	II	III	IV
2	Stream number (N_u)	784	332	68	1
3	Stream length (L_u) (m)	9.33	3.24	0.73	0.007
4	Mean stream length (L_{sm}) (km)	0.012	0.01	0.011	0.007
		II/I	III/II	IV/III	-
5	Stream length ratio (R_L)	0.3472	0.225	0.0095	
		I/II	II/III	III/VI	
6	Bifurcation ratio (R_b)	2.87	4.43	104.2	
7	Mean bifurcation ratio (R_{bm})	37.201			
8	Perimeter (P) (in km)	320			
9	Watershed area (km)	2288.28			
10	Watershed length (Lb) (km)	203.58			
11	Total relief (R) (m)	2184			
12	Relief ratio (R_h)	0.00095			
13	Drainage density (D_d)	0.0058			
14	Stream frequency (F_s)	0.5179			
15	Drainage texture (R_t)	0.1398			
16	Form factor (R_f)	0.0552			
17	Circularity ratio (R_c)	0.280			
18	Elongation ratio (R_e)	0.0003			
19	Length of overland flow (L_g)	85.564			

4. CONCLUSION

In this paper, morphometric analysis of the Mazandran River watershed was conducted based on several drainage parameters using remote sensing data and GIS tools. The study area is mainly dominated by lower order streams. The results show that among the subwatersheds, the highest N_u is 333 (stream order 1 for W1), while the lowest N_u is 1 (stream order 4 for subwatershed W1). For U , among the subwatersheds, W1 and W2 are of order 3 and W3 is of order 4, showing that they are in the early stages of erodibility. W1 has the highest L of streams (5.72 km), whereas W3 has the lowest L of streams (3.11 km). The R_b values for the subwatersheds are between 0.73 to 1.84. D_d varies between 0.0055 and 0.0060 km/km² for the study area. The F_s values for the subwatersheds exhibit positive correlations with drainage density (from 0.48 to 0.58). The minimum and maximum form factors (R_f) are observed for W3 (0.58) and W2 (0.93) respectively. R_c is in the range of 0.43 to 0.75. In addition, the results of geomorphologic analysis show that for all the subwatersheds, the largest landform is streams. On the whole, the analyses show that all three subwatersheds are susceptible to flooding and erosion, and need more management.

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CHEMICAL WEAPONS CONVENTION AND ITS APPLICATION AGAINST THE USE OF CHEMICAL WARFARE AGENTS

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ABSTRACT

The history of the serious efforts to achieve chemical disarmament that culminated in the conclusion of the Chemical Weapons Convention (CWC) began more than a century ago. Although toxic chemicals have been used as a method of warfare throughout the ages, it is clear from some of the earliest recorded incidents that such weapons have always been viewed as particularly abhorrent. In this article a special attention will be paid to each part of the Convention itself, the main aim of the Convention which is the destruction of chemical weapons owned by the signatory states and the control of production, stockpiling and use of chemical for civil use as well. Any violation of the obligations, even inside a state territory, is punished with sanctions provided for in the Convention. The purpose of the article is to understand the obligations that the signatory states are required to observe, the inspection mechanism of specialized organizations, the possible sanctions in case of violations of the obligations verified after the inspections and the future perspectives after the global changes. In this paper the authors will analyze the creation and the evolution of CWC.

Keywords: *Chemical Weapons Convention (CWC), Organisation for the Prohibition of Chemical Weapons (OPCW), Chemical Warfare Agents (CWA)*

1. INTRODUCTION

Before this article can be discussed in greater details, it is necessary to distinguish between warfare means and methods. Warfare means are the weapons or the systems that fighters use on opponents to perform materially violence. Warfare methods are the strategies and tactical procedures used during military operations to overwhelm the opponents using information and weapons effects in addition to surprise. This explanation is important because during the last century, until the adoption of Protocol I in 1977, relating to the Protection of Victims of International Armed Conflicts, fighting interpretations were used to justify the indiscriminate use of weapons of mass destruction with prohibited warfare means and methods. Technical research group like the Quantum Electronics and Plasma Physics Research Group of University of Rome Tor Vergata are working on the problems of non-conventional events by developing new Decision Support System (DSS) (Gaudio *et al.*, 2011, 2013, 2014; Gelfusa *et al.*, 2014; Gallo, 2012; Benedetti *et al.*, 2011; Cacciotti *et al.*, 2014; Di Giovanni, 2014; Lupelli *et al.*, 2014; Malizia *et al.*, 2014; Carestia, 2014, 2015; Cenciarelli, 2015a; Ciparisse, 2015) and analyzing particular events using state of the art techniques and technologies in order to understand and address the problems (Malizia *et al.*, 2010; Malizia, 2016; Pinna *et al.*, 2011; Paziienza *et al.*, 2013, 2014; Cenciarelli, 2014, 2015b; Ludovici *et al.*, 2015).

The legal and moral prohibition of these weapons was successful in order to stop the race to chemical war. Industrial innovations and new technology developments brought the International Community to start a new process to improve law of war to conform it to military technology innovations. The aim of this work is to analyze the Chemical weapons convention from its origin and until its development and application during the years, showing how it has changed to protect humankind from possible criminal uses of chemical agents from new threats like international terrorism.

2. GENESIS AND HISTORICAL DEVELOPMENT OF CWC

2.1 1899 to WWII

An international peace conference held in The Hague in 1899 led to the signing of an agreement that prohibited the use of projectiles filled with poison gas. The efforts of the twentieth century were rooted in the 1899 Hague Peace Conference. The contracting parties to the 1899 Hague Conventions declared their agreement to abstain from the 'use of projectiles, the sole object of which is the diffusion of asphyxiating or deleterious gases'. Their intentions unfortunately proved futile. The rules of warfare agreed at the Hague Conference and its successor (the 1899 and 1907 Hague Regulations) prohibited the use of poisoned weapons. Nonetheless, chemical weapons were used on a massive scale during World War I, resulting in more than 100,000 fatalities and a million casualties (Zanders, 2002).

The result of this renewed global commitment was the 1925 Geneva Protocol for the Prohibition of the Use in War of Asphyxiating, Poisonous or Other Gases, and of Bacteriological Methods of Warfare. However, the Geneva Protocol does not prohibit the development, production or possession of chemical weapons. It only bans the use of chemical and bacteriological (biological) weapons in war. Furthermore, many countries signed the Protocol with reservations permitting them to use chemical weapons against countries that had not joined the Protocol or to respond in kind if attacked with chemical weapons. Since the Geneva Protocol has been in force, some of these States Parties have dropped their reservations and accepted an absolute ban on the use of chemical and biological weapons (Thakur& Ere, 2006).

During the first half of the twentieth century, many developed countries spent considerable resources on the development of chemical weapons, particularly after the discovery of powerful nerve gases which renewed interest in the field. A number of countries used chemical weapons in the inter-war period, and all the major powers involved in World War II anticipated that large-scale chemical warfare would take place. Contrary to expectations, however, chemical weapons were never used in Europe in World War II. The reasons are uncertain, and historians still debate whether it was fear of retaliation in kind, the level of protection of enemy troops, or moral reasons that deterred their use (Bassiouni, 2008).

2.2 WWII to 1990

The fate of some of the stockpiles built up in anticipation of World War II is also uncertain. Many chemical weapons were abandoned, buried or simply dumped at sea. In any event, following World War II, and with the advent of the nuclear debate, several countries gradually came to the realization that the marginal value of having chemical weapons in their arsenals was limited, while the threat posed by the availability and proliferation of such weapons made a comprehensive ban desirable. At that time, the issues of chemical and biological weapons disarmament were linked to each other. Both issues became the subject of active consideration when, in 1968, Sweden was able to include them on the agenda of the multilateral Geneva disarmament conference. At that time, the conference was called the Eighteen Nations Disarmament Committee (ENDC) (Kellman, 2015).

Shortly thereafter, the negotiations on biological and chemical weapons issues diverged. In 1969, the United Kingdom tabled a draft biological weapons disarmament treaty. After several modifications

that reduced its effectiveness, the draft Biological Weapons Convention was agreed upon in the disarmament conference, and was endorsed by the United Nations General Assembly. The treaty opened for signature in 1972 and entered into force in 1975. The Biological Weapons Convention was an incremental step forward in the commitment to achieving a chemical weapons ban. Each State Party to this Convention affirms the recognized objective of effective prohibition of chemical weapons and, to this end, undertakes to continue negotiations in good faith with a view to reaching early agreement on effective measures for the prohibition of their development, production and stockpiling, and destruction. In addition, appropriate measures concerning equipment and means of delivery specifically designed for the production or use of chemical agents for weapons purposes are necessary (Graham, 1992).

The issue of chemical weapons was therefore retained on the agenda of the Geneva Conference, and various states tabled drafts during the 1970s. This era also saw the announcement of a joint US–Soviet initiative on chemical weapons, which was to be submitted to the Geneva Conference. A US–Soviet working group set up during this period began discussing some key ideas, which eventually formed the building blocks of the CWC. These included the need to control the precursors of chemical weapons, to establish mechanisms such as a conference or committee of all states parties and a secretariat to oversee the implementation of the treaty, and to use routine and challenge inspections as part of the verification regime (Raicevic, 2001).

In 1978, the Geneva conference -it was renamed the Conference on Disarmament in 1980- was restructured. Its membership increased to 40, and the chairmanship was to rotate among the members. The Conference decided in March of that year to establish an ad hoc working group on chemical weapons, which was required to 'define, through substantive examination, issues to be dealt with in the negotiations' on the Convention. During 1984, significant developments in the elaboration of the draft Convention have been made. The United States submitted a new draft text, which proposed intrusive verification measures, including mandatory challenge inspections. The negotiations received a new impetus when the Secretary-General of the United Nations announced that chemical weapons had been used by Iraq in its war against Iran. The Conference then agreed to begin elaborating a ban on chemical weapons, and mandated the ad hoc working group accordingly. The group worked based on a 'rolling text' of the Convention on which areas of consensus and disagreement were reflected (Stock *et al.*, 1996).

Beginning in 1986, the global chemical industry actively participated in these negotiations. Unlike the BWC, the negotiators of a chemical weapons ban reached an understanding that this ban would be subjected to international verification. To this end, trial inspections of both industrial and military facilities were undertaken, starting in late 1988. With the thawing of relations between the United States and the Soviet Union, there were a number of major breakthroughs in the negotiations on the Convention. In August 1987, the USSR indicated its willingness to accept, and even extend, the proposals for an intrusive verification regime contained in the 1984 US draft treaty. In the meantime, photographs of a chemical attack on civilians in Northern Iraq in March 1988 were widely published in the media. The international community reacted with repugnance against this use of Chemical Weapons, and within the Conference on Disarmament, the momentum for the conclusion of negotiations increased. In September 1989, President George Bush announced the new US position to the UN General Assembly: instead of total verifiability, the United States would seek 'a level of verification that gives us confidence to go forward'. In 1990, the United States and the Soviet Union also signed a bilateral agreement on chemical weapons, under which the two countries agreed not to produce chemical weapons, to reduce their stocks of chemical weapons to 20% of current holdings; and to begin destruction in 1992. It was also agreed that neither country would have more than 5000 tons of chemical agents by 2002. This agreement never entered into force; it nevertheless marked a willingness on the part of the two major possessors of chemical weapons to work together to eliminate this class of weaponry (Marauhn, 2016).

While the differences between the Americans and Soviets appeared to be diminishing, other issues gained prominence. Several Arab countries, for example, linked chemical disarmament to progress on

nuclear disarmament. Developing countries were generally concerned about whether the Convention would carry any benefits for them. Various new provisions were therefore developed for inclusion in the text of the Convention during the final years of the negotiations, such as:

1. assistance to victims of chemical weapons use,
2. the exemption of some sectors of the chemical industry from routine inspections,
3. the imposition of obligations on States Parties in relation to abandoned chemical weapons,
4. promise on the part of several developed countries -known collectively as the 'Australia Group'- to review export controls and other barriers to trade in chemicals (Üzümçü, 2016).

2.3 1992 to Present

On the whole, the last 'concession' from the industrialized countries, embodied in Article XI on economic and technological development, was probably the key to obtaining broad support for the Convention, since for a number of developing countries free trade in chemicals for purposes not prohibited under the Convention was the only important issue. The solution that was found is perhaps best captured in the statement of the Australian representative to the plenary session of the Conference on Disarmament on August 6, 1992. "*The members of the Australia Group undertook to review, in the light of the implementation of the Convention, the measures that they will take to prevent the spread of chemical substances and equipment for purposes contrary to the objectives of the Convention*". The aim was removing such measures for the benefit of States Parties to the Convention acting in full compliance with their obligations under the Convention (Paragraph 40 of the Report of the Ad Hoc Committee on Chemical Weapons to the Conference on Disarmament, CD/1170, dated August 26, 1992(OPCW, 2016).

In 1992, another obstacle to agreement on the Convention was removed when the United States renounced its previous insistence on retaining the option of retaliation in kind, and accordingly dropped its demand for the right to retain security stockpiles. There was a strong push to conclude the CWC. This was affirmed when President George Bush called for, and obtained agreement on, a one-year deadline for the completion of negotiations. It was clear to everyone involved that 1992 offered a window of opportunity for agreeing on a text for the treaty. The Chairman of the ad hoc Committee on chemical weapons moved quickly and tabled a draft Convention which incorporated the latest 'rolling text' and possible compromise solutions. After two more revisions, the draft convention was approved by most delegations, and was transmitted to the Conference in the summer of 1992. The Conference on Disarmament adopted the draft text on September 3, 1992 and transmitted it in its Report to the UN General Assembly. The text of the Convention was commended by the General Assembly in December 1992, with the request to the UN Secretary-General, as Depositary of the Convention, that it be opened for signature in Paris on January 13, 1993. A total number of 130 States signed the Convention within the first two days and it was subsequently deposited with the United Nations Secretary-General in New York (Üzümçü, 2016).

Recognizing that considerable preparations were required, and that a number of outstanding issues still remained to be resolved before the Convention could enter into force, the signatory states in Paris approved a resolution -the 'Paris Resolution'- to set up a 'Preparatory Commission' for the future Organization for the Prohibition of Chemical Weapons. Under the General Assembly resolution commending the text of the Convention, the UN Secretary-General was also requested to provide the services required by the signatory states to initiate the work of the Preparatory Commission. Accordingly, the Paris Resolution mandated the UN Secretary-General to convene the Preparatory Commission for its first session within 30 days of the fiftieth signature of the Convention. Since this threshold, number was easily exceeded at the signing ceremony in Paris, the inaugural session of the Preparatory Commission was held shortly thereafter, on February 8, 1993, in The Hague, the Netherlands, and the seat of the future Organization. As mandated in the Paris Resolution, the Preparatory Commission immediately established a Provisional Technical Secretariat to assist its work, and to prepare for the eventual Secretariat of the OPCW. The Preparatory Commission stayed in existence from 1993 until shortly after the Convention entered into force on April 29, 1997.

According to the terms of the Convention, the CWC would enter into force 180 days after the 65th country ratified the treaty. To prepare for the treaty's entry into force and the implementation of the verification regime, a Preparatory Commission was established in 1993 (Herbach, 2016).

The work of the Preparatory Commission, as described in the Paris Resolution, was to prepare the 1st Session of the Conference of the States Parties after Entry into Force, to make all necessary practical preparations for the implementation, and to finalize the work and the necessary procedures and guidelines needed for its implementation. These activities can be broadly categorized as developing the operational procedures for the Chemical Weapons' (CW) Verification regime and other operations; drafting the program and budget of the OPCW; and establishing the infrastructure and internal functional rules for the OPCW Secretariat. The Preparatory Commission functioned primarily through two working groups, one of which was tasked with considering administrative and organizational matters, while the other was assigned the responsibility for issues relating to verification and technical cooperation and assistance. Other bodies were also created to assist the work of the Preparatory Commission on specific issues such as relations with the host Country and preparations for the First Session of the Conference of The States Parties. The Preparatory Commission was successful in resolving a number of tasks within its mandate, the results of which were reflected in its Final Report. Among its major achievements were solutions to several substantive verification issues as well as the setting up of the OPCW Laboratory and Equipment Store, the development of a general training scheme for inspectors and the recruitment of inspector trainees, arrangements relating to the new OPCW headquarters building, and the development of draft documents such as the Headquarters Agreement. In addition, there are also an OPCW Staff and Financial Regulations, OPCW Health and Safety Policy and Regulations, OPCW Confidentiality Policy, and the OPCW Media and Public Affairs Policy. The Preparatory Commission was also responsible for the orderly transfer of its property, functions and recommendations to the OPCW. Despite its considerable efforts, however, the Preparatory Commission was unable to reach an agreement on a number of issues deriving from the Paris Resolution. These issues were therefore carried over to the OPCW as 'unresolved issues'. Many of these issues have been resolved since then, but others are still under discussion by the Member States of the OPCW (Herbach, 2016; OPCW, 2016).

Hungary was the 65th country to ratify the Convention, in late 1996, and on April 29, 1997, the Chemical Weapons Convention entered into force with 87 States Parties—becoming binding international law. An additional 22 countries had ratified the treaty in the 180 days between Hungary's ratification and entry into force. With the entry into force of the Convention, the OPCW immediately began its work to implement the Convention. Both, the Convention and its implementing body, are intended to adapt not only to shifts in the international environment and the changing needs of State Parties, but also to respond to the rapid pace of scientific and technological developments. Every five years, the Convention foresees that the State Parties should undertake a review of the implementation process. These review conferences serve as fora for the assessment and evaluation of the CWC's implementation, and the identification of areas where change is needed. A particular focus is given to the verification regime and the changing context within which it is implemented as well as scientific and technological advances in chemistry, engineering and biotechnology. The first review conference was held from April 28 to May 9, 2003. The second review conference was held from April 7 to 18, 2008.

3. STRUCTURE OF CWC AND ITS USE AGAINST TERRORISM

The Convention on Prohibition of the Development, Production, Stockpiling and Use of Chemical Weapons and on their Destruction is an international treaty under which prohibition of chemical weapons is fully regulated. It took over 25 years to complete all the negotiations. The Convention consists of a Preamble, 24 articles and 3 annexes (Iannotti *et al.*, 2016).

3.1. Basic Obligations and Rights of Contracting Parties

The basic obligations and rights of the contracting parties may be classified as (Boulden, 2014):

1. *Obligations related to chemical weapons*: basically the contracting parties are prohibited to develop, produce, acquire, stockpile or keep chemical weapons or transfer them to whomsoever, directly or indirectly;
2. *Obligations related to chemical weapon production facilities*: the contracting parties must officially provide all the data on them to allow the inspectors to complete the phase of inspection and get final decisions;
3. *Obligations related to old and abandoned chemical weapons*: the obligations related to old CWA are not strict as the ones for other CWA.
4. *Obligations related to activities not prohibited under the Convention*: States can use toxic chemicals for purposes different from war or terrorism but they have to provide detailed information about these activities.
5. *Right to support and protection from chemical weapons*: The state contracting party that is under attack with chemical weapons is protected in such situation by this right (the details are in Article X of the Convention).
6. *Right to unfettered economic and technological development and protection of confidential information*: The chemical industry under the CWC has to respect the limitation on production and transfer of certain chemicals, the submission of data on production and the permission to effect inspection on spot.

3.2. Measures Implemented in the Convention

The measures implemented in the convention at (Üzümçü, 2014):

1. *National level*: the provisions to implement these measures are in Article VII of the Convention. These are general provisions that have to be analyzed and used by each state to examine concrete measures in case of CW possession or production. There is not a general model that can be applied for each state.
2. *International level*: the CWC has been established to guarantee an international control on chemical weapons that has to follow different protocols compared to other threats like nuclear weapon production. In order to achieve this, the OPCW -Organization for the Prohibition of Chemical Weapons- has been established together with sanctions, limitations or suspensions of right that can be imposed against the states that violate the provisions of the convention.

3.3. CWC Against Terrorism

There is a large number of CWC provisions that can be used as effective tools to prevent terrorist purposes. Here the important ones are analyzed (Dewing, 2014):

1. *“The Convention requires states to enact laws criminalizing the production of or attempted production of chemical weapons”*: Many states still do not have laws criminalizing the production of chemical weapons. Japan, for instance, did not have such a law before the terrorist incident in 1995. The CWC specifically requires states to enact laws criminalizing the production, stockpiling, transfer, and use of chemical weapons by any persons or corporations on their territory or subject to their jurisdiction. -including persons holding their citizenship, worldwide.
2. *“The Convention requires states to control the production of chemical weapons”*: The CWC requires governments to collect data on a substantial number of chemical weapons and precursors (listed in the CWC's Schedules of Chemicals), providing a ready-made source of data for national anti-terrorist efforts.
3. *“Chemical industries will be alerted to the danger that their products may be misused”*: Some, but not all, chemical industries in some developed countries are already aware of the

need to be cautious in selling chemicals that can be used to produce chemical weapons. The CWC's reporting requirements, combined with national programs of industry outreach, will help to alert firms to the need to use caution in selling precursor chemicals.

4. *“National and international agencies will be created that can serve as resources in the fight against terrorism”*: The creation of the OPCW helps all the states with chemical terrorism potential problems. CWC also requires that the states create a national agency that has to communicate with OPCW all the information about chemical weapons or chemical weapon precursors.
5. *“States will be discouraged from assisting or protecting chemical terrorists”*: The CWC will make it more difficult for states to aid chemical terrorists and their supporters. First, the treaty will reinforce the international norm against the possession or use of chemical weapons, and so expose states that assist terrorists to severe international criticism. Second, it will require states to enact legislation criminalizing attempts to produce chemical weapons. This will remove the excuse that a person who is being sought for crimes elsewhere cannot be extradited if he or she has not committed a crime under the laws of the state in which he or she has taken shelter.
6. *“The CWC will assist states that are the victims of actual or threatened chemical terrorist attacks”*: The CWC provides for humanitarian and technical assistance to states that have been the victims of actual or threatened use of chemical weapons.
7. *“National stockpiles of chemical weapons that might otherwise fall into the hands of terrorists will be eliminated”*: The Convention requires that these stockpiles be destroyed; until this process is complete, they will be under international supervision, reducing the danger of diversion.
8. *“The CWC provides a forum for discussing chemical terrorism-related problems”*: The OPCW's components will include an executive council that can address problems on an emergency basis, as well as annual meetings of the Conference of Parties at which the treaty's operation can be reviewed and adjusted.

4. THE INSPECTION SYSTEM OF THE CWC: PROBLEMS AND PROSPECTS

The CWC has been the first treaty that totally prohibited and eliminated a whole category of weapons of mass destruction with an extremely extensive and intrusive verification system. The Convention has also become a model for subsequent disarmament treaties. In this section the inspections system will be deeply analysed to understand how it applies the control and disarmament treaties of CWC.

4.1 The Significance of a Challenge Inspection System

The CWC provides for two sets of mechanisms to deal with possible concerns about non-compliance:

1. The clarification procedure that clarifies questions concerning possible non-compliance, either bilaterally or through the Executive Council of the Organization for the Prohibition of Chemical Weapons -CWC, Article IX, paragraphs 1–7.
2. The challenge inspection system that allows for inspectors of the Technical Secretariat of the OPCW to conduct an on-site inspection on the territory or any other place under the jurisdiction or control of a State Party when another State Party has raised a concern about non-compliance. If a challenge inspection could establish that there had been no breach of the Convention in all the dubious cases, it would help enhance confidence among States Parties that obligations under the Convention had actually been complied with by others. Even if that were not the case, a strong possibility of detecting breaches could still in itself assure States Parties that obligations under the Convention have generally been met.

It seems that, for the deterrent function to work effectively (ie. have a challenge inspection system), certain conditions should be met:

- a) there ought to be the possibility that inspections can be in fact be conducted at any time - conditions for effective decision-making-
- b) there ought to be the possibility of actually detecting non-compliance -conditions for effective detection-
- c) In assessing whether and, if so, to what extent these requirements are fulfilled, the following elements seem to be relevant:
 - 1) the kind of information that would be required;
 - 2) how a decision on such a request would be made;
 - 3) whether there is a right of refusal on the part of the challenged state;
 - 4) whether there is any quota or limit to the request or receiving of inspections;
 - 5) whether there are any restrictions in terms of specifying inspection sites; the timeline between the decision to conduct an inspection and its actual implementation; and the intrusiveness of inspection activities.

4.2 The Challenge Inspection System Under the CWC

The basic provision of the CWC concerning challenge inspections – Article IX, paragraph 8 – is as follows: “Each State Party has the right to request an on-site challenge inspection of any facility or location in the territory or in any other place under the jurisdiction or control of any other State Party for the sole purpose of clarifying and resolving any questions concerning possible non-compliance with the provisions of this Convention, and to have this inspection conducted anywhere without delay by an inspection team designated by the Director-General [of the Technical Secretariat] and in accordance with the Verification Annex.”

The above-cited paragraph states that the inspections are to be conducted “in accordance with the Verification Annex” of the Convention so an analysis on the provisions of the Annex is necessary to understand the power of this inspection system.

1. *Decision-making on conducting an inspection:* The requesting State Party is first required to submit an “inspection request” to the Executive Council of the OPCW -where the decision on the challenge inspection would be made- as well as to the Director-General of the Technical Secretariat with all the requested information. The inspection request can be refused -with the so-called red-light formula- but it is a rare case and it happens when the request is based on ridiculous motivations. The inspection request is accepted with the three-quarter blocking majority of all members of the Executive Council -the votes of absent persons are counted as “favourable to the inspection”-. There is no quota or limit system applicable to the number of inspections that a State Party or a facility may receive (Cooper, 1992).
2. *Inspection procedures:* The inspected State Party is allowed to take measures to protect sensitive installations and to prevent the disclosure of confidential information and data “not related to this Convention” -Article IX, paragraph 11(c).
3. *Timeline:* The inspection team, once it has arrived, shall assess the “inspection perimeter” proposed by the State Party and evaluate whether to accept it or not. The access to the “requested perimeter” by the inspection team has to be given within 108 hours of their arrival. It should be noted, however, that the inspected State Party must be provided with information regarding the location of the requested inspection site at least 12 hours before the inspection team’s arrival at the point of entry -Verification Annex, Part X, paragraph 6-. Thus, the inspected State Party would have at maximum roughly 120 hours -five days- to prepare for the inspection. The inspection team is also allowed to commence such activities as taking wipes, air, soil or effluent samples within a 50 meters band around the outside of the perimeter upon the team’s arrival at the final/alternative perimeter -“perimeter activities”: Verification Annex, Part X, paragraphs 35– 37-. Both perimeter activities and exit monitoring may be continued until the completion of the inspection -Verification Annex, Part X, paragraphs 31, 35.

4. *Intrusiveness*: The inspection team availability to access in the “requested perimeter” does not mean that the inspection team members are authorized to access all the facilities and areas of the perimeter in order to protect the sensitive installations of the State Party preventing the disclosure of sensitive information. The inspected State Party is obliged to make “every reasonable effort” to demonstrate that any object, building, structure, container or vehicle that has been protected is not used for purposes related to the possible non-compliance concerns raised in the inspection request. (OPCW, 2016).
5. *Assessment*: The Executive Council can allow a semi-automatic decision to conduct an inspection, by the way the inspection modalities present constrains. In terms of timeline, the inspection team might have to wait up to five days before being permitted to enter the requested inspection site. Theoretically, unlimited access would be the best possible way to ensure the effective verification of an arms control agreement but it is not possible: that is why the five days and the access have as a consequence that there is not a 100 per cent verification in a real arms control and disarmament world. The key criterion that is sometimes mentioned in this respect is the ability “to detect militarily significant violations in sufficient time to make an effective response” (Krepon, 1992). However, the concept of the timely detection of militarily significant violation would have different meanings for different arms control agreements. In the CWC context, “one ton of chemical” was once mentioned in the US Senate as a criterion for “militarily significant violation” but it is important to clarify that the concept of “militarily significant violation” or the “one ton” threshold for an effective verification has nothing to do with the definition of chemical weapons themselves in the CWC. Therefore, if a State Party develops or produces a toxic chemical or its precursor beyond the quantity that could be justified in the light of a designated purpose not prohibited under the Convention, it is in breach of the Convention, even if it does not reach a quantity of military significance. Military significance is a concept that could be applied in the context of verification and not in the context of prohibition.

4.3 General Constraints on Conducting Challenge Inspections

The above restrictions are strictly related to the CWC but there are general restrictions that have to be considered. They concern the external and internal functions of state sovereignty: one is related to the territorial jurisdiction or control of a state and the other to the human rights of individuals.

1. *Limitations related to the territorial jurisdiction or control of a state*. Article IX, paragraph 8, of the CWC states that each State Party can request an inspection on another non-State Party. However, normally it is not possible to conduct an on-site inspection on the territory of a non-State Party even if the place is under the jurisdiction or control of a State Party and it might entail a risk of undermining the whole verification system of the CWC. The CWC has introduced the following paragraph in its Verification Annex, Part II to avoid the above-mentioned problem: “In cases where facilities or areas of an inspected State Party are located on the territory of a State not Party to this Convention, the inspected State Party shall take all necessary measures to ensure that inspections of those facilities or areas can be carried out in accordance with the provisions of this Annex. A State Party that has one or more facilities or areas on the territory of a State not Party to this Convention shall take all necessary measures to ensure acceptance by the Host State of inspectors and inspection assistants designated to that State Party. If an inspected State Party is unable to ensure access, it shall demonstrate that it took all necessary measures to ensure access.” The first provision obligates the State Party having facilities or areas on the territory of a non-State Party to take all necessary measures to ensure that inspections of those facilities or areas can be carried out when they are requested. On the other hand, the last provision obliges the State Party concerned to demonstrate that it took all necessary measures if it is unable to ensure access. This latter provision implies that a State Party shall take all measures to satisfy the CWC even if the inspectors cannot have access to the suspected areas. In order to face this problem the CWC has introduced another paragraph that is analogous to paragraph 20 quoted above. Paragraph 21 of the Verification Annex, Part II, stipulates as follows: “In cases where the facilities or areas sought to be

inspected are located on the territory of a State Party, but in a place under the jurisdiction or control of a State not Party to this Convention, the State Party shall take all necessary measures as would be required of an inspected State Party and a Host State Party to ensure that inspections of such facilities or areas can be carried out in accordance with the provisions of this Annex. If the State Party is unable to ensure access to those facilities or areas, it shall demonstrate that it took all necessary measures to ensure access. This paragraph shall not apply where the facilities or areas sought to be inspected are those of the State Party". The last sentence of this paragraph does not appear in paragraph 20. This sentence, in fact, means that the possible exemption from ensuring access stipulated in the paragraph would not apply where the facilities or areas sought to be inspected belong to the hosting State Party.

2. *Limitations related to the human rights of private persons.* The on-site inspections have to take into account constitutional provisions on human rights regarding privacy (Bothe *et al.*, 1998). In order to reduce the possibilities to have a negative answer in term of access in some particular facility, the CWC stipulates in paragraph 41 of the Verification Annex, Part X that: "the inspected State Party shall be under the obligation to allow the greatest degree of access taking into account any constitutional obligations it may have with regard to proprietary rights or searches and seizures" This means that the inspected State Party is required to give the inspectors access only to the extent that this is in conformity with its constitution. Therefore, even if the inspected State Party cannot obtain a warrant and give full access -in time-, it would not be held to be in breach of the Convention for failing to give such access (Duncan *et al.*, 1995).

4.4 Practical Problems of the Challenge Inspection System

The non-use of the challenge inspection system and its background, arguably the most challenging problem of the CWC's challenge inspection system, has been that it has been neither used nor requested since the last years causing a negative effect on the credibility of CWC. It was due to the difficulties to prove the presence of chemical weapons that still exists, even if the technologies and methods to detect chemicals have been improved in the last decade. The second problem is that a non-detection does not mean a "non presence" of chemical weapons or chemical weapons precursors, so that an innocence declaration can be affected by errors. Developed States Parties, on the other hand, put emphasis on the difference between the clarification procedure and the challenge inspection system, and maintain that the clarification procedure is not something that is legally required to be followed as a precondition before a challenge inspection request is filed. They point out that the provision in Article IX, paragraph 2, itself makes that point clear by stating that "States Parties should, whenever possible, first make every effort". This is to clarify and resolve non-compliance concerns through consultations and that the clarification procedure is to be utilized "without prejudice to the right of any State Party to request a challenge inspection". Legally speaking, the point of view of developed States Parties reflects the correct interpretation of the relevant provision. Nonetheless, the argument of developing States Parties is not necessarily a complete reinterpretation of the above provision and seems to contain some legitimacy. At any rate, the difference of opinion between the two groups appears to be rather profound and it does not seem that it will be settled anytime soon. Perhaps there therefore needs to be a fresh approach to accomplishing the goal.

4.5 Towards Resolving the Problem

A mechanism developed that was proposed to overcome the above mentioned problems affirm that "there seems to be a need to create another level of mechanism which falls between the routine industry inspection and the politically loaded challenge inspection. A mechanism which is a purely technical exercise but which serves to clarify questions and uncertainties which delegations and the Organization may have. Such a mechanism, denuded of a political character, could serve a useful role as a confidence building measure that goes beyond the provisions of the regular inspection" (OPCW, 2000).

The fact that the challenge inspection might not work as expected had been anticipated even during the CWC negotiations. In 1989, to counter such an occurrence, the United Kingdom had proposed an “ad hoc inspection” system with rather limited purposes and scope. These ad hoc inspections have to follow these lines:

1. Each State Party would have the right to initiate inspections by the Technical Secretariat in civil and military facilities and elsewhere on the territory of any other State Party.
2. These requests would not be linked to any allegation of breach of the Convention.
3. The purpose of the inspection would be to check whether any activity in the facility concerned was subject to declaration or prohibition under the terms of the Convention.
4. Procedures for the conduct of the inspection (i.e. its format) would differ from those for routine inspections and for challenge inspection. This proposal was not accepted during the CWC negotiations because developing countries saw it as being too similar to the challenge inspection and, in addition, they did not wish to receive further inspections at their industrial sites (OPCW, 2016).

However, a similar system was proposed and agreed upon in the nuclear field. In May 1997, the Board of Governors of the International Atomic Energy Agency –IAEA- adopted a model Additional Protocol (INFCIRC/540) to the model Comprehensive Safeguards Agreement (INFCIRC/153). The latter Agreement has provided a basic mechanism for monitoring the nuclear activities of non-nuclear-weapon States Party to the Treaty on the Non-Proliferation of Nuclear Weapons –NPT-. It is called “routine inspection” based on reports prepared by those States. In 1991, the IAEA revealed the problems connected to this system because clandestine nuclear weapon programs have been found in States like Iraq (Albright & Hibbs, 1992). Faced with this reality, the IAEA decided to formulate a new set of rules to monitor nuclear activities.

If the CWC had been able to adopt an “ad hoc inspection” system, the problem that the CWC is now facing with regard to its challenge inspection system might not have come about. Put differently, the problem might well be resolved, at least partially, if the OPCW could adopt, and States Parties could accept, a document comparable to the Additional Protocol of the IAEA. Thus, the idea contained in the Protocol’s “complementary access” system seems worth exploring in the CWC verification context. The difficult part is how to achieve the goal. From a methodological perspective, there appears to be two ways to introduce such a system into the CWC. One is to utilize the existing framework; the other is to create a new framework. The first option might draw on the precedent of confidence- and security-building measures –CSBMs- in Europe. The CSBMs in Europe, a mechanism designed mainly to promote transparency by providing information on military activities, are equipped with a challenge-type inspection, called “inspection”. According to the Stockholm CSBM Document of 1986, an “inspection” may be requested when compliance with CSBMs is in “doubt”; and an inspection request needs to be accompanied by a statement of “reasons” for the request (Bloed, 1993). However, these conditions and requirements were later dropped in the Vienna CSBM Document of 1994 (Bloed, 1993), probably owing to the routine of inspection requests in practice. It would be tempting to follow this example of European CSBMs and drop the sensitive part of the information requirement in requesting a challenge inspection in the CWC context -i.e. concern regarding possible non-compliance-. Yet the reality would not be so simple. First, unlike the European CSBMs, which are based on a series of evolving political documents, the CWC is a legally binding treaty. As such, it is impossible to drop an important requirement for an inspection request without formally amending the Convention, which is far more difficult than modifying CSBM Documents. Secondly, the modification of the European CSBM Documents became possible because the participating states routinely made requests for inspection, which is something completely lacking in the OPCW. A second method to achieve the goal would be to negotiate a new document on complementary access-type inspections in the Additional Protocol of the IAEA. However, it would be equally difficult to pursue this path. One needs to remember that States Parties to the CWC have already assumed a considerable burden in receiving industry inspections every year, depending on the scale of the respective States Parties’ chemical industry. It is inconceivable that they would assume a

new burden without being offered any new carrot. The same factor seems to have led to the dismissal of the “ad hoc inspection” proposal during the CWC negotiations. To agree on any new measures, the minimum requirement would be a general agreement among participants to promote the shared idea, which is again lacking in the OPCW at present. This train of thought brings us back to the method of utilizing the existing framework. It is possible to envisage a State Party requesting a challenge inspection of the facilities of another State Party that has friendly relations with the requesting State Party, thus breaking the ice. Admittedly, this could be seen as an irregular, if not abusive, request, but it might still be regarded as falling within the scope of the CWC challenge inspection scheme, as long as the request is for the purpose of “clarifying and resolving any questions concerning possible noncompliance with the provisions of this Convention” -Article IX, paragraph 8, emphasis added-, no matter how technical the questions may be. In other words, the language of the CWC concerning challenge inspection seems broad enough to cover not only challenge inspections proper but also the “complementary access” type of inspections. It is to be hoped that such an “evaluative” interpretation would promote a practice that could be followed by other interested States Parties and gradually constitute a basis on which to build a system similar to the “complementary access” of the IAEA.

5. THE THIRD REVIEW OF THE CWC

The OPCW area and goals have changed during its history and among the traditional goals of the regime are the complete, irreversible and verified destruction of existing CW stockpiles, which still needs to be accomplished, and the continued oversight of the regime’s non-proliferation dimension— or, as it is increasingly called, the prevention of the re-emergence of chemical weapons. These two areas have been characterized as the ‘fundamental goals’ of the CWC by the US Under Secretary of State for Arms Control and International Security, during the UN high-level meeting. The interventions by Iranian and other representatives at the same meeting on behalf of the Non-Aligned Movement –NAM- have highlighted and reasserted a wider set of goals and different priorities, focusing on international cooperation, assistance and protection. These new goals together with the experience of the first two CWC Review Conferences in 2003 and 2008 have been the base to implement the third revision of the CWC as it is a mistake to think that the CW problem has been solved with the establishment of the CWC (Lachowski, 1994; Robinson, 2008).

The main focus of the CWC implementation has been the destruction of existing CW stockpiles by possessor states, which has led to approximately 75 per cent of declared CW having been destroyed under international verification by 30 June 2012. The six CW possessor states — Russia, the United States, India, South Korea, Albania and Libya — have declared a total of nearly 70,000 tons of chemical warfare agents and about 8.6 million munitions and containers. Of these, Russia declared some 40,000 tons, the United States 28,575 tons, India around 1,000 tons and South Korea around 600 tons. Both the Albanian and the Libyan declarations—some 16 tons of CW agents in the case of the former and 23.62 tons in the case of the latter—did not substantially change the overall size of declared CW stockpiles. The OPCW fixed in the past a ten-year deadline – April 29, 2007-to complete destruction of declared CW stockpiles, with a five-year extension option (OPCW, 2016). However, it has not been respected, considering that the US in 2006 had destroyed less than 50% of its CW arsenal, India around 70%, South Korea more than 80%, and the Russian Federation around 16%.

These delays resulted in the extension of the final destruction deadline for five of the CW possessor states, which was combined with a requirement to report to the Executive Council every 90 days on the progress made in the destruction process, as well as to continue to submit annual plans of destruction and annual reports on their CW destruction activities. It has been decided to set visits intended as ‘additional transparency and confidence building measure’. After these measures, positive results have been achieved: in summer 2012, the US had destroyed approximately 90 per cent and the Russian Federation 65 per cent of their respective declared CW arsenals, raising the potentially thorny

issue of how the Review Conference will deal with the delay in CW demilitarization (Lachowski, 1994).

5.1 Non-Proliferation and Preventing

After the above-mentioned results, the budget agreed at the end of 2012 allocates fewer resources to the inspection of CW destruction activities and to implement a higher number of inspections of so-called 'other chemical production facilities' –OCPF (Kelle, 2012).

The majority of the chemistry substances used to produce chemical weapons have dual use application to rules and procedures for toxic chemicals that may pose a risk to the CWC's object and purpose, but are not listed on one of the three CWC schedules, they are called DOC -Discrete Organic Chemicals-. The verification of these OCPFs has been a cause of disagreement among CWC states parties during the past 2 decades. Since the CWC entered into force, more than 5000 inspected States Parties have declared DOC-producing OCPFs. The United States during the years stressed the need to increase the number of OCPF facilities 'that are inspected annually' and to focus more on 'specific facilities that should be inspected'. Some of these facilities incorporate technologies and features that are highly relevant to the Convention (Kelle, 2012).

There are several diverging views on OCPF inspections so in accordance with CWC the OPCW had to start the implementation of a verification system for OCPFs. In the first stage, the site selection for inspections was carried out through a two-stage process in which first the country and then the plant site for inspection were selected. An interim algorithm introduced by the Technical Secretariat in May 2007, which allowed for the selection of plant sites in a single step and sought to direct the process towards relevant facilities, replaced this temporary mechanism (Kelle, 2012). The improved algorithm allows the Technical Secretariat to focus on facilities of greater relevance but does not satisfy the part: 'proposals by states parties'. A mechanism for how such proposals could be integrated into the OCPF site selection methodology still needs to be agreed upon more than a decade after OCPF inspections were begun.

5.2 Protection and Assistance

The protection against the CW is legitimate according to article X. Such protection can be realized through research, development, production and use of protective measures against CW. In order to improve the protection capabilities, many exercises and training courses have been conducted. The first major OPCW exercise on the delivery of assistance, named ASSISTEX¹, took place in September 2002 in Zadar, Croatia. Its aim was to assess the preparedness of both states parties and Technical Secretariat for processing and responding to a request for assistance. The underlying scenario involved a fictitious state party discovering a terrorist group first producing and then using CW in an attack on a major airport. Over 900 individuals from eight states parties participated in the exercise. It has been followed by several exercises that help the implementation of CWC provisions on protection, assistance thanks to the multitude of activities by both the OPCW's Technical Secretariat and a small number of dedicated states parties, which have funded, conducted and provided logistical support for a large number of assistance- and protection related courses. It has clearly improved the capabilities to conduct an investigation of alleged use and to provide assistance if the national capabilities of a CWC state party are unable to cope with the situation.

5.3 International Cooperation for Peaceful Uses of Chemical Substances

In order to attract those states that have never produced CW or do not feel threatened by them, the CWC contains provisions -in article XI- for fostering international cooperation in the peaceful uses of

¹<https://www.opcw.org/our-work/assistance-protection/>

chemistry. The OPCW's Technical Secretariat has developed and implemented a range of activities in this area, annual sessions of the Conference of the State Parties and the first two review conferences have seen members of the NAM criticizing the export controls of states participating in the Australia Group –AG-, which they regard as contravening the CWC cooperation provisions. The number of states participating in the AG has grown and the scope of its export controls has widened since then, while criticism has recently become restricted to a few NAM states (Robinson, 1992).

Some of these States strongly criticize the existence and operations of AG so the members of the Western European and Others Group –WEOG-, by contrast, defended the need for the Australia Group's continued existence. Given the entrenched positions of participants in this debate, it is not surprising that much of the Review Conference report simply reproduced text contained in the CWC or agreed upon during earlier conferences of the state's parties. The Review Conference also 'urged the Council to continue its facilitation efforts to reach early agreement on the issue of the full implementation of Article XI' (Robinson, 1992).

6. CONCLUSION

The aim of this work has been to analyse the CWC starting from its origin, its development and application during the years, showing how it has changed to protect humankind from possible criminal uses of chemical agents coming from new threats like international terrorism. The study has focused on an historical analysis on the evolution of chemical weapon production and use in different countries and how, year by year and case by case, new procedures, laws and approaches have been developed and used to face these problems and increase the safety of people and security of countries till the preparation of CWC.

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THE LOCAL EFFECTS OF A GLOBAL DISASTER: CASE STUDY ON THE FUKUSHIMA RADIOLOGICAL EMERGENCY MANAGEMENT IN ITALY

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ABSTRACT

CBRNe events that occur in a geographic location can have consequences on human health and/or infrastructures, even in places very far away from that where the event happened. Thus, the effects of such events often have an influence on the response systems of various countries, from a socio-economic point of view, from where the event had its origin. In this work, the authors dealt with the evaluation of the consequences of the accident at the Fukushima (Japan) nuclear power plant and in particular with the management of the effects of this event in a European country (Italy). The study was focused on the clinical examination of 50 people coming back to Italy from Japan in the days immediately following the disaster that occurred at the Fukushima nuclear power plant as an effect of the tsunami. During the screening, external and internal contaminations were checked; thyroidal monitoring was carried out to estimate the exposure to Cs and I. For all the 50 monitored persons, the measured values of contamination were not higher than the attention value, so the basic level analyses were sufficient, without the need to do further and more thorough examinations.

Keywords: *Chemical, biological, radiological, nuclear & explosives (CBRNe); Fukushima nuclear power plant; health protocol; monitoring; scintillation counts.*

1. INTRODUCTION

The Fukushima power plant accident, which happened as a consequence of the seismic event of 11 March 2011 has been one of the worst in history: the global relevance of the event has been clear since the first phases (Chino *et al.*, 2011) and has reached the seventh degree in the INES (International Nuclear and Radiological Event Scale) scale of the International Atomic Energy Agency (IAEA) (IAEA, 2008). Unlike

the Chernobyl disaster of 1986, in Fukushima, there was no direct damage to the reactor and thus, there was no huge emission of transuranic elements in the environment.

The diffusion of radioactive nuclei in the environment has mainly concerned cesium and iodine (Katata *et al.*, 2012; Terada *et al.*, 2012), whose effects have been measurable on a global scale (for example by the European net) (Masson *et al.*, 2011). As a result of the great media interest, one of the indirect consequences of the event has been called “iodine rush” (Durigon & Kosatsky, 2012), i.e. a rush to purchase iodine doses to use as a preventive prophylaxis to contrast the accumulation of radioactive iodine in the human body. This rush happened although the excessive consumption of iodine by a healthy person was not recommended by World Health Organization (WHO), as it can cause serious health problems (Pennington, 1990).

The Fukushima accident, because of its geographical location, has been made global because it has involved a highly industrialized and interconnected country, so the people coming from other nations, when they returned to their homeland, weighed on the local health systems. The Fukushima accident is an example of how a chemical, biological, radiological, nuclear and explosive (CBRNe) event can have a global relevance and several local effects. The preparedness in the management of such emergencies cannot be abstract. In addition to technical points of view (Malizia *et al.*, 2012, 2014; Gallo *et al.*, 2013; Cacciotti *et al.*, 2014; Sassolini *et al.*, 2014; Di Giovanni *et al.*, 2014, Pirelli *et al.*, 2014), psychological perspective is also important, as the recent Ebola epidemic has demonstrated (Cenciarelli *et al.*, 2014, 2015a, 2015b).

The aim of this work is to illustrate the protocol concerning the management of the Italian citizens coming back from Japan in the weeks which immediately follow the accident, and to report and study the results of analyses that were carried out.

2. MATERIAL AND METHODS

2.1 Control Protocol

On 25 March 2011, the Italian Ministry of Health regulated the care pathway for people coming back from Japan (Ministry of Health, 2011). The activities, carried out by Reference Centers, were monitored in collaboration with the National Institute of Health, in order to harmonize and rationalize the response. The procedures were developed to respond to citizens that voluntary went to the hospitals previously identified by the Regions as reference centers.

In the document issued from the Italian Ministry of Health, three situations were considered, depending on the distance from the area where the people had stayed/transited to the epicentre of the disaster: a) distance greater than 80 km; b) distance less than 80 km; c) close to the epicentre. It is important to recall that the recommendations of WHO, at the moment of the publication of the document, foresaw the need of controls after the return from Japan exclusively for those who had stayed/transited in the surroundings of the power plant and inside the evacuation zone (20 km) (Ministry of Health, 2011).

In case (a), the protocol does not foresee radiometric or dosimetric examinations on the person, but it just suggests giving information and an adequate psychological support. The latter tends to reduce anxiety by means of a correct communication on the absence of an exposure to radioactivity higher than that to the natural one.

In case (b), the protocol foresees the gathering of information and an instrumental evaluation of the eventual exposure, by following these steps: 1) Taking charge using the structure individuated by the Health Direction (for example, nuclear medicine, first aid, etc.); 2) Gathering of information on: place of origin, duration of permanence in the area, time elapsed since the re-entry into Italy (to be excluded if longer than 30 days), eventual data on thyroidal and renal activity, alimentary habits, eventual ingestion of iodine or medicine against thyroidal pathology, and eventual recent medical treatments by administration of radioactive nuclei; 3) Carrying out experimental survey. The results of these controls will then be communicated to the National Health Institute (ISS, *Istituto Superiore di Sanità*) for the formation of a national information archive.

In case (c), which should already have been treated by the local authorities, the protocol foresees to gather more detailed information about the place, the exposure duration and the modality of staying in the surroundings of the nuclear power plant, and about what happened between the period during which the person stayed in that area and the request of controls in the hospital. Further instrumental examinations can be done. In cases where the results of these medical surveys indicated a risk for health, the recovery in a hospital and the start of appropriate medical treatment will be initiated.

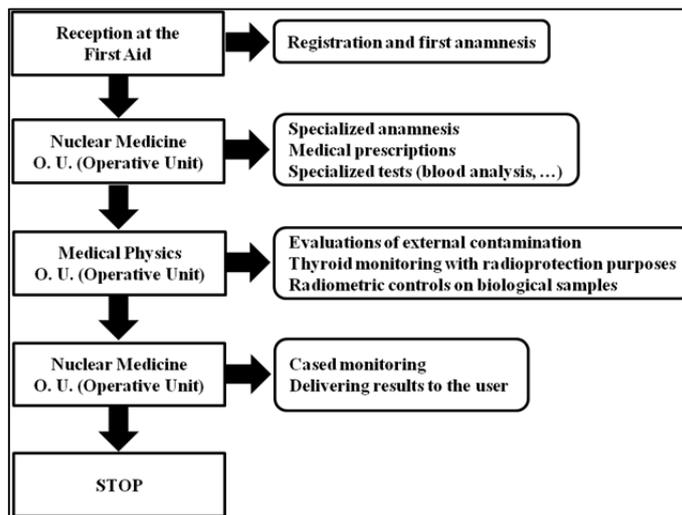


Figure 1: Path scheme for returning people from Japan with suspected radioactive contamination.

2.2 Instrumental Measurements

The radiometric measurements suggested by the protocol follow the guidelines elaborated in the frame the IDEAS Project (EU 5th Framework Programme) (Doerfel *et al.*, 2006) for the estimation of the absorbed radioactivity dose on the basis of individual monitoring data. The guidelines are based on three fundamental principles: harmonization, accuracy and proportionality. Following these principles, the guidelines use measurement levels to estimate the yearly taken dose, considering values ranging from 0.1 to 6 mSv.

Each measurement level is characterized by specific procedures. At the laboratory where this work was carried out, in order to evaluate the measurement level, the measurements described in the paragraphs below have been done.

2.2.1 Verification of the External Contamination

Measurement of the dose rate in contact with the whole body; for the verification of the external contamination, in Antero-Posterior (AP) and Postero-Anterior (PA) projections, an environmental ionisation camera Inovision Victoreen, model 451P_DE_SI_RYR s/n 009 (Fluke) was used.

2.2.2 Thyroidal Monitoring

The measurement of the absorption by the thyroid with an external scintillation probe was done to estimate the exposure to Cs and I by the recording of the scintillation counts obtained by a Atomlab 950 Biodex Medical System s/n 1194013 system, which is constituted by a NaI(Tl) scintillator.

In both cases, a measurement of the environmental radioactivity has been done in the measurement room, with the following settings:

- Window for ^{131}I (309-420 keV) with the probe set at 20 cm from the thyroid and with an acquisition time equal to 5 min;
- Window for ^{137}Cs (560-759 keV) with the probe set at 2 m from the patient sitting down and with an acquisition time equal to 5 min.

As foreseen by the 0 Level of the IDEAS guidelines, it has been verified that the yearly absorbed dose does not exceed the reference value of 0.1 mSv. To this end, in the case of the measurement of the counts performed with the Atomlab 950 system, a procedure to convert the number of counts in the taken dose was applied.

Thus, the constancy of the conversion ratio between the number of counts and the radioactivity was calculated. By a range of measurements of the activity of known sources, the following conversion value from counts per second (cps) to activity (Bq) was estimated:

$$6,091 \text{ cps} \rightarrow 37 \text{ KBq} = 37,000 \text{ Bq} \quad [1]$$

Based on this:

$$A(\text{Bq}) = \frac{37000}{6091} \text{ cps} = 6.07 \text{ cps} = 0.101 \text{ cpm} \quad [2]$$

After having done that, the absorption curve of ^{131}I versus the thickness of a Plexiglas sheet put in front of a source whose activity is known was determined. Plexiglas is considered as a material tissue-equivalent. Considering that the mean depth of the thyroid can be estimated to 1.75cm, the attenuation value of 0.75 can be obtained from the curve shown in Figure 2.

Taking into account the attenuation value (= 0.75), the following conversion value was obtained:

$$A(\text{Bq}) = \frac{0.101}{0.75} \text{ cpm} = 0.135 \text{ cpm} \quad [3]$$

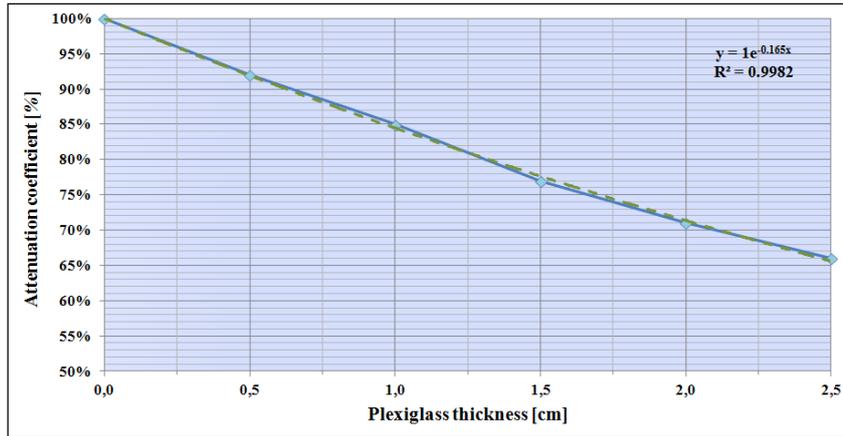


Figure 2: Absorption curve of ^{131}I versus thickness.

The yearly effective dose was calculated from the expression:

$$\sum_j h(g)_{j,ina} J_{j,ina} \quad [4]$$

where:

$h(g)_{j,ina}$ is the committed effective dose per introduction of the radio nucleus j ($\frac{\text{Sv}}{\text{Bq}}$) that is inhaled by a person belonging to the age group g and $J_{j,ina}$ is the introduction by means of inhalation of the radio nucleus j (Bq).

3. RESULTS AND DISCUSSION

In this section, the results of the radiometric measurements carried out in March 2011 on 50 people in Italy coming back from Japan are presented. The results of the verification of the external contamination using an ionisation camera are reported in Figure 3. The maximum measured value of external contamination is equal to 0.6 $\mu\text{Sv/h}$ and the normalization from the background values does not exceed, except in a few cases, the background level itself. In those rare cases where the difference was greater than twice the background value, the measures were not considered to be high enough to proceed to a phase of more thorough analysis.

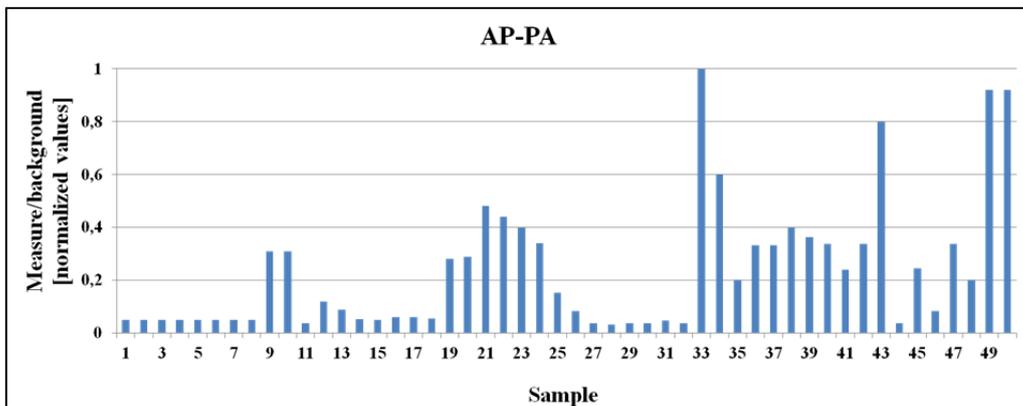


Figure 3: Ratio between external contamination and environmental radioactivity (normalized values).

The results of the thyroidal counts for the ^{131}I estimated annual dose with respect to the background are shown in Figure 4. The measured values are practically comparable to the ones caused by the natural background. In the case of the measurement with the whole body in order to estimate the contamination from ^{137}Cs , on the basis of the obtained results for thyroid, the measures with difference with respect to the background were analysed and a reference threshold of 1.5 times the background was set. In Figure 5, it is shown that this value has never been exceeded.

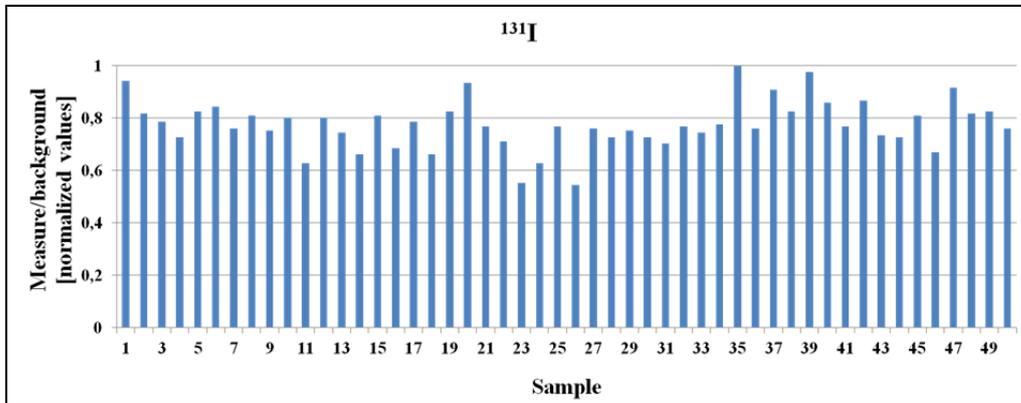


Figure 4: Ratio between the estimated annual taken dose of ^{131}I and the one due to natural background (normalized values).

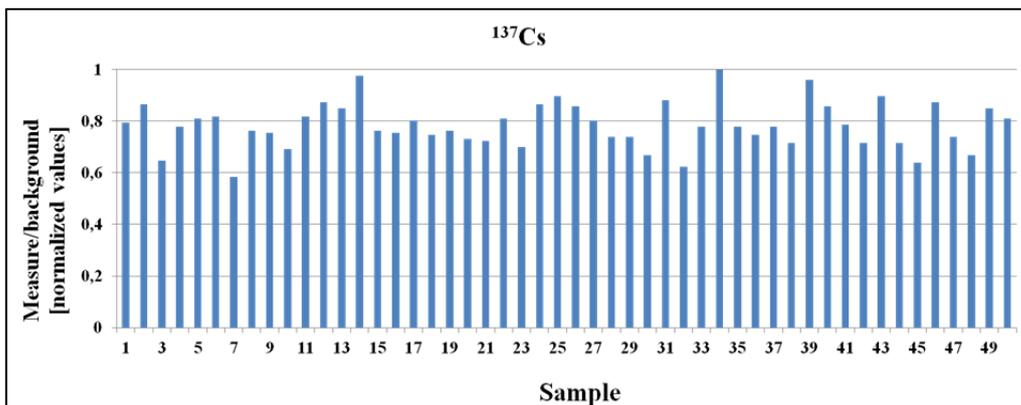


Figure 5: Ratio between the number of scintillation counts per minute measured for ^{137}Cs and the ones due to background.

4. CONCLUSION

In this work, the monitoring activity carried out by a medical physics laboratory in Italy after the Fukushima disaster in Japan has been presented. As shown from the results, for all the 50 monitored people, the measured values of contamination were not higher than the attention value, so the basic level analyses have been sufficient, without the need to do further and more thorough examinations. However, in the opposite case, as foreseen by the IDEAS guidelines, more accurate examinations would have been done with methods such as gamma spectrometry of urine and faeces, which would have made it possible to evaluate in a more precise manner the absorbed dose. In the case that it would have been necessary, on the basis of the results obtained in the second phase, models and mathematical expressions with the corresponding values of the parameters as suggested by the IDEAS guidelines will be used, in order to satisfy the fundamental harmonization principle.

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