

## **Tribological and Mechanical Performance of Epoxy Reinforced by Fish Scales Powder**

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<https://doi.org/10.18280/rcma.320306>

### **ABSTRACT**

**Received:** 3 April 2022

**Accepted:** 14 June 2022

#### **Keywords:**

*fish scales powder, tribological behaviour, mechanical performance, epoxy composites*

The study was conducted to use fish scales powder as animal biomass to prepare epoxy composites. Fish scales powder is beneficial in reducing environmental pollutants. The fish scales powder was added to the epoxy matrix for improving the interfacial bonding between the scales and the epoxy matrix. However, a direct method was used to prepare epoxy composites, and samples were cut according to ASTM standards for mechanical and tribological tests. Interfacial interaction between the fish scales powder and epoxy was investigated by FTIR and SEM. It was found that the fish scales powder contents affect the mechanical properties and tribological behaviour of produced composites. Compared to pure epoxy, the load of 10 wt.% fish scales powder increased the tensile strength by 16.0%. As well as, the coefficient of friction was reduced by 16.0% and wear resistance was enhanced by 48.58%. The improvements in the performance of composites are contributed to the hydrogen bonding formed between fish scales powder and epoxy matrix.

## **1. INTRODUCTION**

Developing high-performance epoxy resin-based materials is finding an increasing number of applications, which require high tribological performance to reduce energy consumption. Reducing friction is one of the keys to energy management. Because of friction, the annual energy loss is estimated to be 30% [1, 2]. Simultaneously, with the downsizing and integration of devices, energy usage has greatly increased [3]. Since epoxy has low cost, high thermal stability, excellent chemical and resistance to humidity [4, 5] as well as good adhesive and mechanical performance, which made it is mostly selected for floorings and airport runway repairs as a protective coating against severe wear [6-8].

However, the inherent poor wear resistance of cured epoxy seriously restricted tribological applications [9]. To overcome this issue, several types of fillers were used, such as silica, alumina, and clay, as well as, graphene oxide, graphene nanoplatelets and multi-wall carbon nanotube are added to the epoxy matrix [10, 11]. The composites reinforced by nano-fillers such as ceramics and carbon-fillers have several critical disadvantages such as several manufacturing complications, poor adhesion to the epoxy matrix and non-biodegradability. As a result, these materials depend principally on depletable resources, resulting in rapid environmental hazards.

Recent critical reviews about natural resource protection and recycling have led to renewed interest in biomaterials such as sisal [12], palm and jute fibers [13-16]. However, many researchers have mentioned a considerable improvement in the performance of composite fibers reinforced by plants fibers caused by the weak interfacial bonding between the hydrophobic polymers and hydrophilic plants fibers like palm fibers and jute sisal [17]. However, there has been an increasing interest in natural fillers around the globe, mostly

due to the fact that they are environmentally beneficial and have a low price. Also, bio-fibers such as animal hairs, bird feathers, and fish scales have recently drawn the researchers' consideration [18].

Various useful materials are composed in fish scales including trace elements such as calcium, zinc, iron, magnesium and 38-46% inorganic components (calcium - deficient hydroxyapatite, calcium phosphate, etc.) and 41-45% organic components (sclerotin, fat, lecithin, vitamins, collagen, etc.) [19-23]. Additionally, the mechanical performance, like tensile modulus and ultimate tensile strength of fish scales, was calculated as around 2.2 GPa and 93 MPa, respectively [9]. The good mechanical features of fish scales motivated researchers to use them to strengthen an epoxy matrix to improve the epoxy matrix mechanical properties. Collagen is the main component of fish scales which is an organic protein [24]. This causes the conclusion that bonds of hydrogen can be created between the epoxy oxygen elements and the polypeptide chain's hydrogen elements in collagen structure when the fish scale is used as a reinforcement [25]. As a result, there is no need for chemical modification of fish scale using chemical reagents as it could cause break down the structure of the fillers causing to reduce its performance of strengthening the epoxy matrix.

Regrettably, to our knowledge, the effect of fish scale particles on the tribological performance of epoxy composites is rarely studied, so the purpose of this research is to investigate the tribological behaviour of fish scale reinforced epoxy composites with different weight fractions using a direct mixing process to improve its tribo-properties. The bonding and compatibility of our composite could be enhanced by mixing the epoxy resin with a fish scale, such as presenting reaction sites with larger chemical affinity with the surface of the fillers. This could lead to our goal resulting from

the strong interfacial interaction between epoxy matrix and fish scale. The unique potential of using this method is its attractive price-performance ratio and easy operation potential that helps it become the most widely used epoxy composite for tribological applications.

## 2. EXPERIMENTAL

### 2.1 Materials

Epoxy resins (Euxit 50KI) with the curing agent (Euxit 50KII) were supplied by Egyptian-Swiss Co., Ltd. Iraqi freshwater Carp fish was used in this study as shown in Figure 1.



Figure 1. Iraqi fresh water carp fish

The Fish Scale powder (FSp) with a size up to 200  $\mu\text{m}$  was prepared in the laboratory. Tap water was used to wash Ripe fish scales to remove soluble surface impurities and adherent dust. Then the scales were dried in the oven at a temperature of 70 degrees Celsius for one day to grind and turn them into powder. Figure 2 shows the fish scales before and after grinding process.

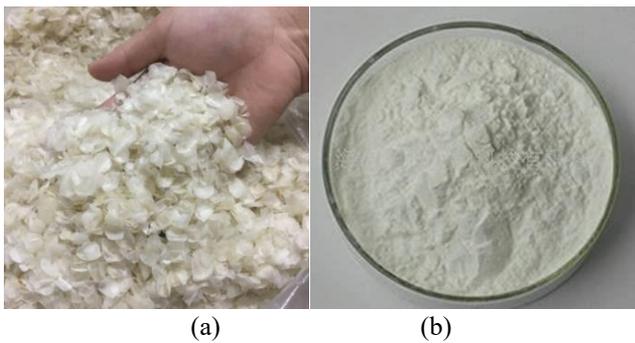


Figure 2. Fish scales a) before grinding. b) after grinding

### 2.2 Preparation of fish scales powder /epoxy composites

Fish scales powder with loading 0.5, 10 and 15 wt.% by high shear mixing was dispersed homogeneously in epoxy resin for 5 min at ambient temperature followed by high amplitude ultrasound for 30 min. Subsequently, the hardener was added to the fish scales/epoxy and mixed on a magnetic stirrer at room temperature for 10 minutes under a vacuum to eliminate air bubbles. Finally, pouring the mixture into Teflon molds and curing it for a week at room temperature. Figure 3 explains the technique for the preparation of fish scales powder -epoxy composites.

### 2.3 Characterization and testing

The elements of the surface chemistry of fish scales and

their composites were examined by Fourier infrared spectroscopy (Bruker, Tensor-27 - Germany). The dispersion of fish scales in the treated epoxy was seen by a light microscope (MEIJI TECHNO CO. Ltd - Japan) [26, 27]. It was studied using a field emission scanning electron microscope (Inspect-S50, FEI CO. Ltd) [28-31]. Tensile performance of composites was performed on a universal testing machine (Beijing United test Co. Ltd) [32-34] with a speed of 2 mm/min, according to (ASTM D-638).

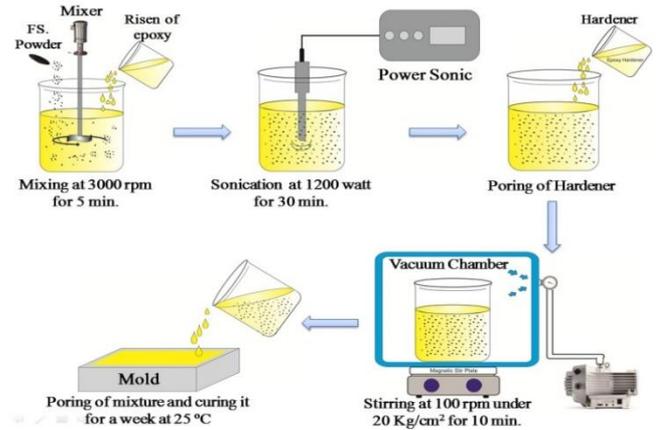


Figure 3. Schematic demonstration of the method adopted to prepare fish scales powder/epoxy composites

The flat type specimen has been used for the tensile test. The dimensions of a specimen are (20, 3, and 165) mm with a dog bone shape. Five separate magnitudes were made to provide the mean value and deviation. The hardness test was performed onshore D hardness tester (Landtek) and achieved as per ISO-868 with dimensions of 5 mm thickness and 50 mm diameter. The tribological test of the coefficient of friction and wear resistance test was achieved using vertical friction and wear testing machine MMW-1A (Beijing united test Co. Ltd - Chain) and was conducted by ASTM G-99. Figure 4 shows the scheme of the test area.

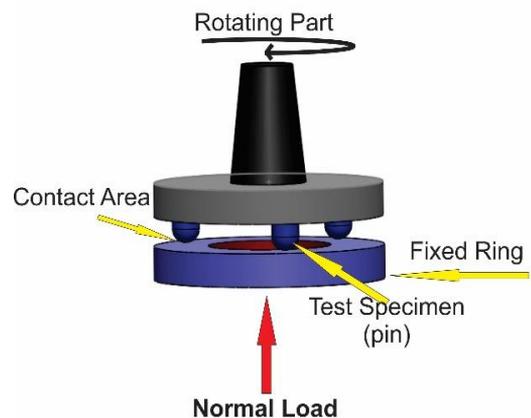


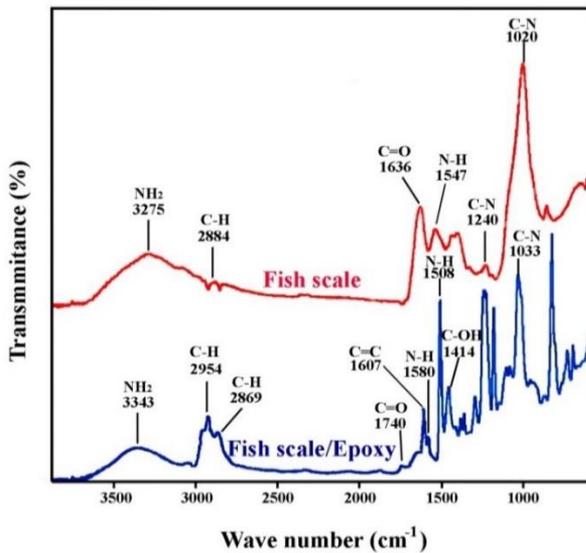
Figure 4. The schematic of the tribological test area

## 3. RESULTS AND DISCUSSION

### 3.1 Structure analysis of the fish scales powder and its composite

As shown in Figure 5, the Fourier-transform infrared spectra (FTIR) was adopted to detect the chemical groups of

fish scale and fish scales/epoxy composite. The broadened peak at  $3275\text{ cm}^{-1}$  in scale is mainly from N-H stretching vibrations. The peaks of C-H and C=O, N-H and C-N originating from the amide group (I, II and III) are observed at 2884, 1636, 1547 and  $1240/1020\text{ cm}^{-1}$ , respectively. After adding fish scales to the epoxy matrix, the peak of N-H and C=O from the fish scale is noted at 3343 and  $1740\text{ cm}^{-1}$  [35, 36]. Therefore, in the composite the newly formed hydrogen bonds between O-H can be confirmed. Epoxy and fish scales interact via the bonds of hydrogen formed between the epoxy chain's oxygen and the polypeptide chain's hydrogen [25]. In addition, the presence of the amino group in fish scales can give better wettability with epoxy resins that can improve the interfacial interaction between the matrix and the filler. The interfacial reactions taking place between the fish scales and epoxy resin are schematically illustrated in Figure 7.

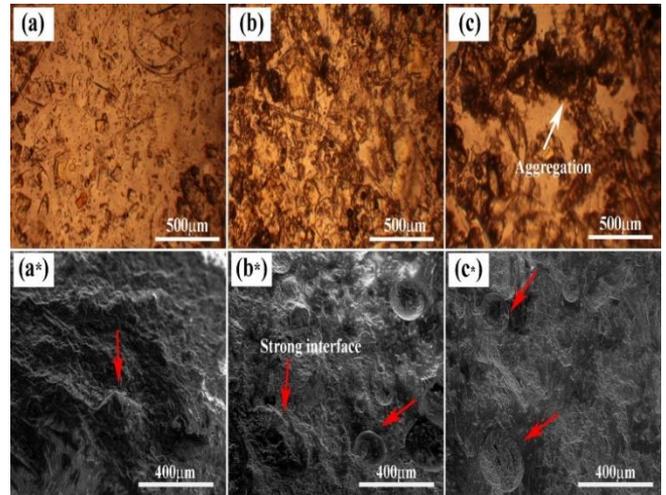


**Figure 5.** FTIR spectra of fish scales and fish scales/epoxy composite

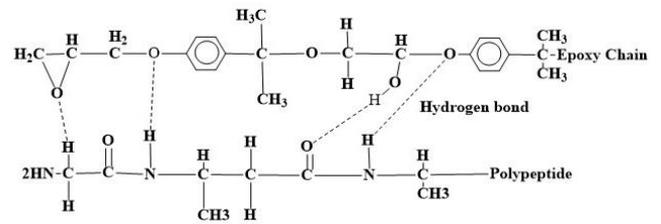
### 3.2 Reinforcing analysis

To explain the fish scales contribution to the fish scales/epoxy composites' tribological performance, the interfacial interaction between the epoxy and fish scales and the dispersion state of fish scales in epoxy were studied by SEM and optical microscope, respectively. The fish scales with a loading of 5 wt.% take up less space than the scales with a loading of 10 and 15 wt.%, as shown in Figure 6a. The fish scales composites were prepared from scales with a loading of 10 wt.% and had a more uniform distribution within the epoxy matrix (Figure 6b). The fish scales occupied more space inside the matrix than the matrix reinforced by scales with loading 5 and 15 wt.%. The high loading of fish scales powder with the matrix tended to accumulate (Figure 6c); which could reduce the epoxy liquid's ability to cover homogeneously the fish scales. Besides, the weak dispersion of the fish scales within epoxy could cause reducing in the mechanical properties and wear resistance of the composite. The interfacial affinity of fish scales/epoxy composites from the fracture surface morphology was confirmed, as shown in (Figures 6a\*, b\* and c\*). The epoxy composite fracture surface reinforced with 5, 10 and 15 wt.% had homogeneous micro-cracks, ascribing to the transfer of stress via the fish scales. The micro-cracks coalescence and crack deflection resulted in a rough surface.

When the cracks reach the fish scales particles, the fracture energy is dissipated [10], which enhances the tribological performance of the composites.



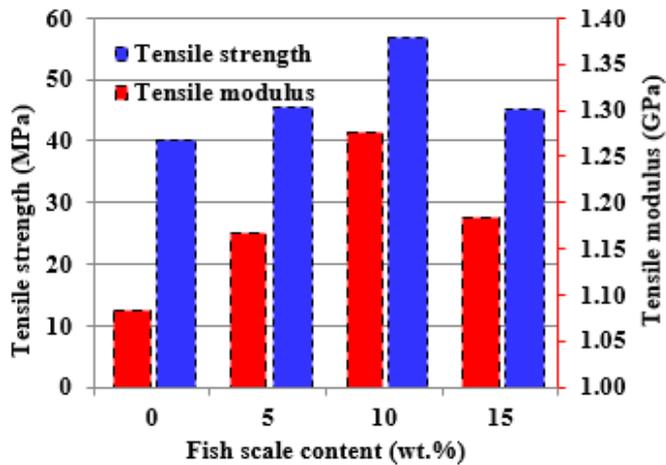
**Figure 6.** Microscope and SEM images of (a, a\*) 5 wt.%, (b, b\*) 10 wt.%, (c, c\*) 15 wt.%



**Figure 7.** Chemical interaction between chain of polypeptide and the epoxy resin elements

### 3.3 Tensile test

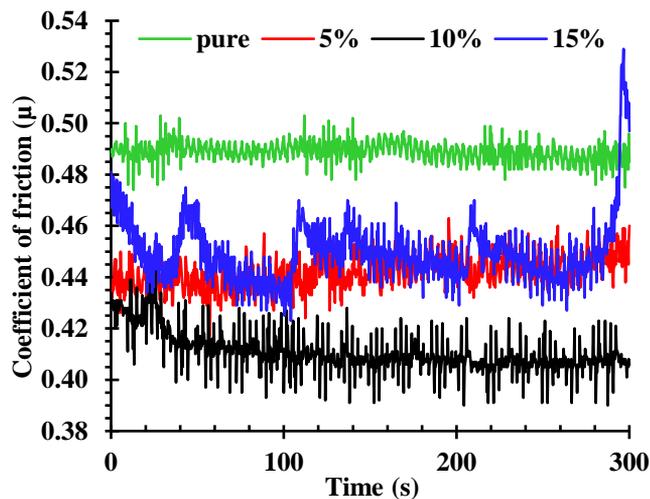
Young's modulus and Tensile strength of epoxy and its composites are illustrated in Figure 8. The pure epoxy tensile strength is 40.2 MPa. In contrast, the epoxy matrix reinforced by 5, 10, and 15 wt.% of FSp increased the tensile strength to 45.5, 46.7, and 45.2 MPa, respectively. The pure epoxy shows Young's modulus of 10.83 GPa, whereas 5, 10, and 15 wt.% of FSp show the young modulus of 11.66, 12.75, and 11.83 GPa, respectively. The concentrations of FSp have an apparent effect on epoxy's Young's modulus and tensile strength. The highest tensile modulus and ultimate tensile strength are attaining at the loading of 10 wt.% FSp, which are 17.72 and 16.17% respectively higher than pure epoxy. Tensile strength and Young's modulus have improved because of the bonding between the epoxy matrix and the reinforcement particles. In addition, as shown in Figure 6, when the fish scale content was increased by 10 wt.%, the fillers occupied a large area of the matrix. This could restrict the crack propagation within the epoxy matrix, thus leading to the improvement of the tensile strength and Young's modulus of composites. When the epoxy loading of 15 wt.% FSp a dropping in tensile strength and Young's modulus remarked, which are 3.21 and 12% respectively lower than 10 wt.% FSp. The reduction in the tensile strength happens at the loading of 15 wt.% FSp, which results in weak dispersion of the fish scales powder within epoxy (Figure 6c). The inefficient distribution leads to agglomerations of additives and shear stress concentration under the action of the tensile force [11, 37].



**Figure 8.** Tensile modulus and tensile strength of pure epoxy and its composites

### 3.4 Tribological test

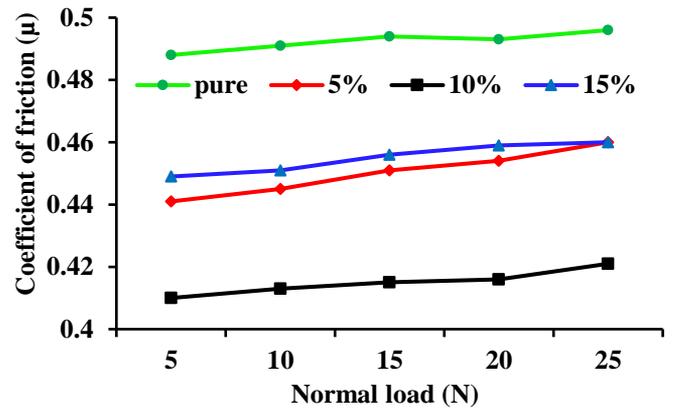
The tribological performances of FSp/epoxy composites strengthened by varying fillers percentages of 0, 5, 10 and 15 wt.% of FSp were investigated. The coefficient of friction behaviour of epoxy reinforced with fish scales powder has been shown in Figure 9. All specimens had been tested in similar environmental conditions and test parameters (spindle speed, normal load, and duration) were 200 rpm, 10 N and 300 seconds respectively. All additive concentrations reduced the coefficient of friction. As observed in Figure 8, the composite of 10 wt.% FSp was given the optimum enhancement in COF as compared with composites filled with 5 and 15 wt.% fish scales powder. Unstable COF behaviour was detected at composite filled with 15 wt.%, the decline is due to the aggregation and stress concentrations at interfacial bonding that leads to the weak stress transfer between epoxy and fish scales powder [38].



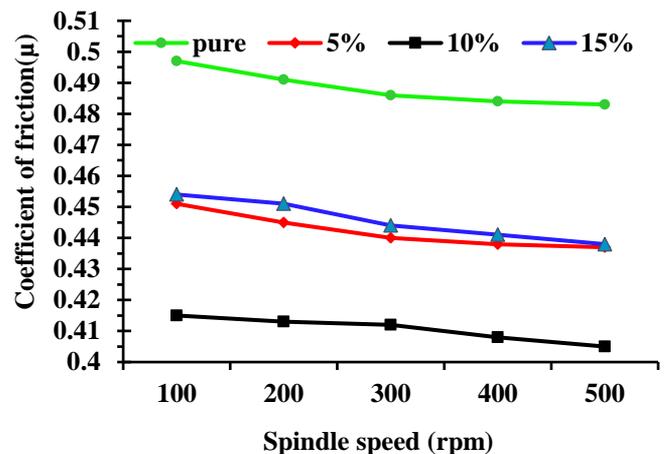
**Figure 9.** Behaviour of coefficient of friction for pure epoxy and its composites at 200 rpm spindle speed and 10N normal load

Figure 10 describes the correlations between the coefficient of friction and normal load. COF of the produced composite was measured by applying load of (5-25 N) at 200 rpm spindle speed for 300 seconds. The COF of epoxy and its composites of (5, 10, 15) wt.% FSp were increased (1.6, 4.3, 2.7 and 2.4)%

respectively. The slightly increased in coefficient of frictions of FSp/epoxy composites with the increases of normal load were due to the increase the interaction of worn surfaces, so the coefficient of friction was also increased [39]. The composite of 10 wt.% FSp was given the optimum enhancement in COF as compared with composites filled with 5 and 15 wt.% fish scales powder. The relationship between the frictional coefficient of composite and spindle speed was shown in Figure 11. The COF measured as a function of spindle speed (100 to 500 rpm) was carried out at a 10 N normal load for 300 seconds. The COF was decreased slightly with increased spindle speed. The increase in disc temperature is a strong function of sliding velocity [40]. In dry sliding behaviors of FSp/epoxy composites the increase in sliding velocity led to elevate disc temperatures [41, 42].



**Figure 10.** The correlations between coefficient of friction and normal load, at 200 rpm spindle speed



**Figure 11.** The relationship between frictional coefficient of composite and spindle speed, at 10 N normal load

The energy released by friction is the primary contributor to the creation of heat, which in turn the contact heat is determined. The contact heat is an essential component that may considerably alter the mechanical characteristics of the FSp/epoxy composite. Additionally, the temperature of the surface that is produced as a consequence of the contact heat can play a major role in the tribo- behaviour of the FSp/epoxy composite. The softened layer low thickness usually causes low coefficient of friction and wear intensity. The adhesion that exists between the composite material asperities and the abrasive ball will deteriorate here at this layer. When shear pressures reach a critical level, FSp/epoxy composite begins

to accumulate in front of the shear stress that produces a plastic flow. This causes the material to be displaced out of the system and break off [43].

The wear resistance of FSp/epoxy composite in terms of volume losses was shown in Figure 12. The volume losses of the composite were measured by applied load (5N to 25N) and weight percentage of fillers, all specimens in similar environmental conditions were tested for 30 minutes to reach the study state behaviour. As a result, adding fish scale powder to the epoxy improved its wear resistance, owing to the formation of a hydrogen bond between FSp and the epoxy matrix. Also, the composite's wear resistance has improved because of the improved interfacial connection between the matrix and the fillers as well as improving the dispersion of fillers within the epoxy. This leads to better stress transfer from epoxy to the fillers [39]. The epoxy matrix filled by 10 wt.% of FSp exhibited the optimum wear resistance for all applied loads.

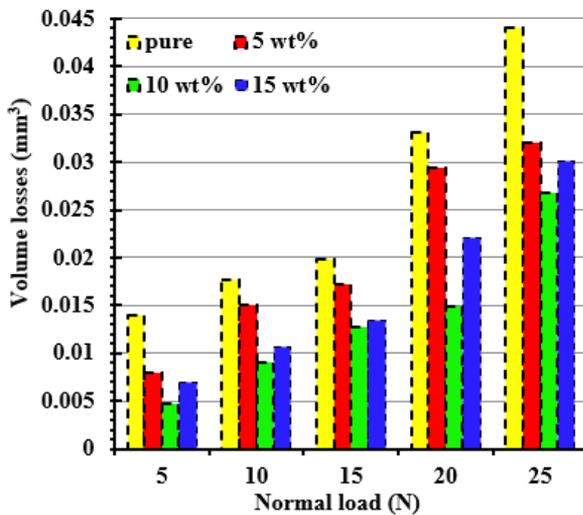


Figure 12. Volume losses vis applied load at 200 rpm spindle speed and 30 min

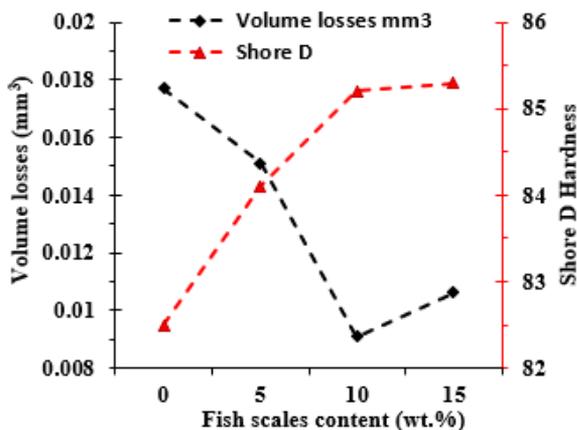


Figure 13. Volume loss and shore D hardness vis fish scale content

In Figure 13 the relationship between wear resistance, additives and hardness was investigated. A volume loss was decreased with increased hardness and additives concentration due to the hydrogen bond between the filler and. At 15 wt.% FSp in epoxy, unstable tribological behaviour was observed (see Figure 9), which is due to increased stress concentrations at the agglomerated fish scales that led to separation of

reinforcement additives with the surrounding area of epoxy, which played an important role in material removal due to dry sliding [44]. If not removed, the particles can protect the polymer [45].

#### 4. CONCLUSIONS

This study is based on the recycling of waste fish scales resources and its application in high-performance epoxy composites, by mixing fish scales powder with liquid of epoxy using a direct mixing method. By testing the tensile, hardness, tribological properties, structural and morphological characterization of the composites, the following conclusions are obtained:

1. It was found that the Young's modulus and tensile strength were improved when adding fish scale powder to the epoxy matrix, resulting from improving dispersion and interface bonding between the fish scales and the matrix within the matrix.
2. At 10 %wt. of fish scales powder combined with epoxy resin results in maximum enhances the wear resistance and coefficient of friction. This is due to the presence of amino-group in fish scales, which promotes the strengthening of the interfacial bond.
3. The highest hardness value has been recorded at the loading of 15 wt.% fish scales powder, which is 85.3 (shore D hardness).

#### ACKNOWLEDGMENT

The authors would like to thank Materials Techniques engineering department labs for their kind support.

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