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Effect of sawdust impregnation on long coir fibers reinforced with epoxy matrix

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ABSTRACT

The purpose of this study is to improve mechanical property of long coir fibers of length 100-150mm using well graded sawdust filler and to scrutinize viability when subjected to distill and sea water conditions for calculated duration. Study involves fabricating eco-friendly composite specimens by maintaining optimum percentage of natural fibers and fillers without compromising on mechanical strength. The composites of different proportions by percentage of matrix (85%, 80%, 75%, 70%), reinforcement (15%, 15%, 15%, 15%), and filler (0%, 5%, 10%, 15%) by mass is developed by hand layup method and compared for their mechanical properties in dry, distilled and sea water conditions. Mechanical properties of coir composite materials improved in addition to sawdust fillers up to certain percentage. In dry condition tensile, flexural and hardness was observed. A maximum tensile and flexural modulus of 7.61Gpa and 2.16Gpa respectively was observed. Higher water absorption was noticed in specimen with higher filler percentage than the specimen with no filler. This is due to hydrophilic nature of sawdust and coir. Specimens immersed in distilled and sea water observed a slump by certain percentage in their strength with increase in fillers. Efficient utilization of filler has improved mechanical performance up to certain percentage when compared to specimen with no filler. As the filler percentage increased above 5% due to the higher fiber length and cluster behavior of coir, resistant in resin penetration was observed as a result specimen turned out to be more and more redundant.

1. Introduction

In composites materials, fibers are the major reason for the strength, whereas matrices maintain the bonding between the fibers and prevent from external harm. Synthetic fiber reinforced fibers have their vast application in multiple disciplines, due to increase in environmental awareness and concern towards global warming; natural fiber polymer matrix composites have gained attention in recent years. The specific properties of natural fibers are ease of availability, low density, Co_2 neutrality and biodegradability. This biodegradability factor results in healthy ecosystem (Anilkumar et al., 2014). Most commonly used natural fibers are plant fibers like coir, jute, sisal, kneaf, banana, hemp, pineapple etc. due to its ease of availability and low cost. The major draw backs of natural fibers are weak adhesion with the matrix material as a result of incompatibility, reduction in mechanical performance and high water absorption rate due to its hydrophilic nature (Rajesh and Ratna Prasad, 2013). The mechanical performance of fibre composites rely on (1) Matrix and fibres properties; (2) content, length distribution and orientation of the fibres in the composite and (3) fibre-matrix bonding that accounts in load transfers (Baiardo et al., 2004). In this study coir fiber composite is developed taking into consideration of all the mentioned dominant factors.

Coir possesses 26-43% cellulose, 0.2% hemicellulose and 41-45% lignin content (Nam et al., 2011). Due to the presence of high lignin content and low cellulose content unlike other natural fibers, treatment is essential for effective interfacial bonding (Haridas, 2014). Fibers are subjected to NaoH pre-treatment process as it removes the lignin content to some extent and other surface impurities.

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This results in increased surface roughening of fiber so as to improve the adhesion between fiber and polymer matrix (Jayabal et al., 2012). Moisture diffusion in polymeric composite is enabled by three mechanisms. The first involves, water molecules diffusing through the micro gaps between polymer chains. The second involves capillary transport through the gaps and flaws at the interfaces between fiber and the matrix. This is a result of poor wetting and impregnation during the initial manufacturing stage. The third involves transport through micro cracks in the matrix arising due to fiber swelling [especially in the case of natural fiber composites] (Espert et al., 2014). Previous study on fiber length shows that as the fiber length increases the tensile strength also develops (Abdul, 2014). The study showed that as the coir fiber length increased up to 15mm, maximum tensile strength of 48 Mpa was observed. Fiber length increased up to 20mm results in decreases in tensile strength due to the curling effect of long coir fiber. The curly nature of fiber results in improper alignment of fiber in composite (Kalaprasad et al., 2004). Contradiction to this study, it was investigated that coir fiber length of 30mm exhibited a higher tensile strength of 13.05Mpa and flexural of 35.42Mpa in coir fibre reinforced epoxy composites and higher strength was due to strong fiber-matrix adhesion so as to transfer higher load (Sandhyarani et al., 2011).

In this study long coir fibers are reinforced with epoxy matrix with sawdust as filler. Sawdust is a major biological waste generated in wood polishing firms, stored in uncontrolled condition can be a factor responsible for environmental pollution (Deac et al., 2016). Variation in chemical composition for sawdust observed was 27.2%-31.7% of lignin, 44.5%-46.65% cellulose, 15.69%-16.29% hemicellulose, 0.01%-0.02% nitrogen content (Mercy et al., 2011). As a concern towards environment, it is very important to prevent pollution resulting from sawdust. In this research sawdust waste is utilized to improve mechanical performance of long coir fiber epoxy composite by replacing proportion of epoxy by sawdust.

2. Materials and method

2.1. Materials

The coir fiber used for the preparation of composites is arranged from local resources. Initially all the coir fibers are segregated finely and they cut into pieces of length about 100-150mm after alkalization. Epoxy resin(LY 556) is used as matrix. To improve thermal and mechanical properties of epoxy resin, curing agent called hardener is mixed with epoxy resin in a ratios 1:10 respectively. The curing agent or hardener utilized is triethylenetetramine (HY-951). Sawdust acquired from local resource is tested for physical properties like specific gravity, particle size, porosity and density.

2.2. Alkali treatment

Coir fibers are alkali treated to modify their surface before fabrication of bio composite (Aly and Nashar, 2016). Long coir fibers were subjected to 6% NaOH treatment for 72 hours as in Fig. 1 to ameliorate the compatibility between the reinforcement and resin. Alkali treatment disrupts the hydrogen bonding resulting in surface roughness by removal of lignin, pectin and dirt contents and also inhibit water absorption rate.

Fig. 1: Alkali treatment of coir fiber

2.3. Fabrication of composite

Composite are fabricated using hand layup method with varying proportions of reinforcement, matrix and filler as shown in Table 1. The hand lay-up process includes steps such as preparation of mold, gel coating, lay-up of materials and finishing (Senthilnathan et al., 2014). Initially petroleum wax is coated on to plywood mold surface for easy removal of composite. Epoxy and hardener by ratio of weight is mixed along with sawdust filler for 10 min. This mixture is laid into the mold of theoretically calculated volume 300 × 300 × 3.2 mm^3. Coir fibers are distributed throughout the mold volume along with remaining epoxy sawdust mixture. The mixture is now pressed under load for uniform distribution of matrix. Composites are then allowed to cure at room temperature for 24 hours.

2.4. Testing of composite

Composite specimens were machined as per ASTM standard and is test for its mechanical properties like tensile strength, tensile modulus, flexural strength, flexural modulus and hardness test at dry condition and ageing (Distilled and sea water) respectively. Water absorption and diffusion coefficient of specimens immersed in distilled and sea water is determined.

2.4.1. Density test

A difference will be observed in actual composite density when compared to theoretical calculated

density due to the presence of void content which accounts in change in mechanical performance. Air void content is calculated using Eq. 1.

$$\text{Void content (\%)} = \frac{Theoretical\ density - Actual\ density}{Theoretical\ Density} \quad (1)$$

Table 1: Proportion of coir fiber, epoxy and sawdust used for fabrication

Composite sample/ Specimen No.	Matrix composition (%)	Reinforcement composition (%)	Filler composition (%)
1	85	15	0
2	80	15	5
3	75	15	10
4	70	15	15

2.4.2. Tensile test

Specimens subjected to tensile test are machined as per ASTM D 638-03 using CNC machine. Tensile test is carried out using universal testing machine at a constant rate of 2mm/min. tensile strength and tensile modulus is calculated using Eq. 2 and Eq. 3 respectively.

$$\sigma t(\text{Mpa}) = \frac{Load\ in\ KN}{Cross-sectional\ area\ of\ specimen(mm*mm)} \quad (2)$$

$$E_t\ (\text{Gpa}) = \frac{Tensile\ strength*Length\ (mm)}{Displacement\ (mm)} \quad (3)$$

2.4.3. Flexural test

The flexural test was performed as per ASTM D790-03 test standards with dimension 12.5 x 125 x 3.2 mm^3. The three point bend test was performed on the composites using same universal testing machine (FIE UNITEK 9450) at a cross head speed of 1mm/min. spam length to depth ratio 16:1 as per standard is considered. Flexural strength and flexural modulus is calculated using Eq. 4 and Eq. 5 respectively.

$$\sigma f\ (\text{Mpa}) = \frac{3PL}{2bt*t} \quad (4)$$

$$Ef\ (\text{Gpa}) = \frac{P*L*L*L}{4*b*t*t*t*d} \quad (5)$$

2.4.4. Hardness test

Matzusawa micro-hardness tester was used to measure the micro-hardness of composite specimens as per ASTM E-384. A diamond intender with an apical angle of 136° was intended over the surface of the specimen under a load of 100gf under dwell time of 15sec. After the removal of load indentation is measured and converted into hardness value.

2.4.5. Water absorption test

Water absorption specimen of size 75mm×25mm×3.2mm was machined as per ASTM D 570 and is immersed in distilled water and sea water at room temperature (27° C). Water absorption percentage was calculated using Eq. 6. Graph of moisture intake percentage Vs time in Hours is plotted and diffusion coefficient (m^2/sec) is calculated using Eq. 7.

$$\text{Water absorption (\%)} = \frac{w2-w1}{w1} \quad (6)$$

$$D(m^2/sec) = \pi x(\frac{B}{4Ms})^2 x(slope)^2 \quad (7)$$

3. Results and discussion

3.1. Physical properties of sawdust

Table 2 shows the physical properties of sawdust filler which play a major role in improving mechanical performance.

Table 2: Physical properties of sawdust

Properties	Numerical value
Specific gravity (IS:2720, Part-3, 1964)	2.67
Density (g/m^3) (Maharani et al., 2010)	0.83
Porosity (%) (Horisawa et al., 1999)	37.3
Particle size (µm) (IS:2720, Part-3, 1964)	<65µ, well graded

3.2. Density test

Table 3 shows density test results. Percentage of void content was calculated as void content in the laminates accelerate water absorption rate. Increased void content was observed due to incorporation of hand laying procedure. Generally tendency of agglomeration is more in long a fiber (Pickering et al., 2016) which is one among the reason for void content. Agglomeration of sawdust at higher filler percentage increased micro pores because of the porous nature resulting in improper curing. Higher void content of 42% was observed in 3rd sample.

Table 3: Void content in composite sample

Sample no	Void content (%)
1	24.6
2	34.5
3	42.9
4	37.4

3.3. Water absorption test

Water absorption specimens of different proportions was machined as per ASTM standard and were dipped in distilled water (pH = 7) and sea water (pH = 8.1). Fig. 2 and Fig. 3 shows weight gain of the composite increased with respect to time at the initial stage and at later stages being stable specifying specimens are saturated with moisture. As the filler increased water absorption increases, increase in filler results in agglomeration which affects the curing of resin matrix. Water absorption percentage of samples increased with increase in sawdust filler up to 10%, as water propagating through cracks and air voids of sample is absorbed

by sawdust and coir fiber due to hydrophilic nature. Coir fibre and sawdust contains cellulose and hemicellulose compositions. These compositions contain various hydroxyl carboxyl groups which might interact forming hydrogen bond with water molecules. Cellulose and hemicelluloses contains hydroxyl group which results in poor interface and offer poor resistance towards moisture absorption. Further the porosity of sawdust (0.37) is the void space in the particle (Baker et al., 1988) occupied by water/moisture enhancing water absorption percentage. Further increase in filler to 15% a decrease in water absorption percentage than 10% filler sample was noticed, as percentage void content is less in 4th sample compared to 3rd sample which inhibited moisture absorption rate of the specimen. The fabrication and machining defects observed in 4th sample was fewer compared to 3rd sample due to improved bonding between fiber and matrix.

At the initial stage of immersion, ions present in sea water accelerate the water uptake through the cracks and voids. Since distilled water is deionized, there are no ions supporting the acceleration of water uptake. Hence Diffusion coefficient of sample immersed in sea water is slightly higher than distilled water and is represented in Table 4.

Water absorption percentage of specimen in sea water is less than that of distilled water due to salt in sea water which is less readily absorbed than water diminishes water molecule action. This result in settling of salt on specimen surface as shown in Fig. 4. The salt concentration in specimen immersed in sea water is less than that of actual sea water as a result an osmotic pressure is created which acts against the water absorption (Zafar et al., 2012).

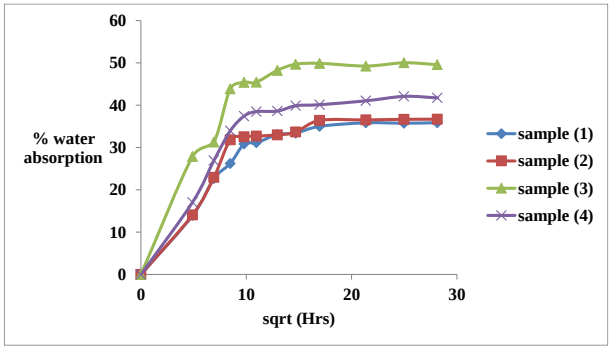

Fig. 2: Distilled water absorbed (%)

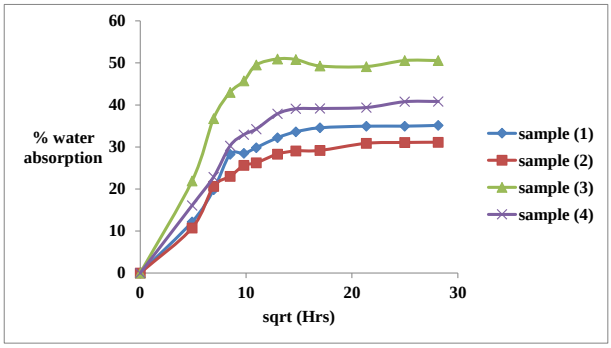

Fig. 3: Salt water absorbed (%)

Table 4: Salt accumulation on specimen surface immersed in sea water

Sample no	Diffusion coeff- Distilled water (m^2/sec)($\times 10^{-12}$)	Diffusion coeff- Sea water (m^2/sec)($\times 10^{-12}$)
1	1.30	1.31
2	1.31	1.27
3	1.60	1.73
4	1.47	1.67

Fig. 4: Salt accumulations on specimen surface immersed in sea water

3.4. Tensile test

Tensile test was carried out using universal testing machine. Fig. 5 and Fig. 6 shows tensile strength and tensile modulus of long coir fibers composite sample respectively. Addition of filler upto 5% increased tensile strength due to uniformity in epoxy matrix distribution i.e., reduced matrix shrinkage and then with further increase in filler a drop in tensile strength was noticed because of poor interfacial matrix-fiber bonding. As the filler increases, matrix is reduced resulting in weaker bonding with the fiber. The result of weak bonding between the water absorbing filler and the water resistant matrix polymer obstructs the stress propagation, and thus reduce the tensile strength when the filler loading increases. In addition, poor dispersion results in filler clouding and drops in tensile properties (Hardinnawirda and Sitirabiatull, 2012). Maximum tensile strength of 13.2Mpa is noticed with 2nd sample i.e., 5% filler. An increase from 8.23Mpa for sample without filler to 13.2Mpa for sample with filler was observed. This due to improved distribution of matrix by the micro fillers allowing it penetrate through the piled up coir fibers. A Large drop in tensile strength was noticed for all the samples immersed in sea water and distilled water. 2nd sample observed a drop from 13.2Mpa to 3.16Mpa and 5.8Mpa when immersed in sea water and distilled water respectively.

The presence of alkaline salts in sea water has resulted in higher degradation of those samples

immersed in sea water than distilled water (Libo and Nawawi, 2015). Higher mechanical degradation in tensile strength by 56% for 2nd samples immersed in distilled water is noticed. This may be due variability in composition (Bessadok et al., 2009). There can be chances that specimen subjected to water immersion had higher voids than overall composite laminate, as voids in the composites need not be uniformly distributed. As the filler content increased mechanical degradation in tensile strength decreased. This must be due to improved fiber alignment and more uniform distribution of epoxy by the filler materials penetrating through the gaps of long coir fiber.

Higher tensile strength degradation for samples immersed in sea water is observed in 4th sample. A reduction by 90.2% is observed. As the filler increased percentage degradation in tensile strength increased due to higher chances of sawdust agglomeration making alkaline salts easier to weaken the matrix interface with filler and fiber. Increased void content in samples results in increased water absorption which results in higher drop in mechanical properties. While Fig. 6 shows the variation of the Young Modulus of the composite at different filler loading. Young modulus is the property that defines the resistance against deformation. Higher the young modulus, lower the tendency of deformation. At 5 wt% filler loading, the Young Modulus showed a remarkable increase. However, as the filler loading continued, a drop in young modulus is observed. Same results had been reported (Aramide et al., 2009).

In addition, the Young modulus of the composites dropped after reaching the Maximum values, which is 7.61GPa. This shows that the stiffness of the composites is contributed predominantly by inherent stiffness of sawdust filler and epoxy matrix (Rozman et al., 2003). Degradation in tensile modulus after immersion in sea and distilled water was observed just as in case of tensile strength. Largest reduction is observed in sea water due to the presence of alkaline salts. Similar results are reported (Libo and Nawawi, 2015). Reduction of threshold tensile modulus to 0.64Gpa and 3.36Gpa was observed for the samples immersed in sea and distilled water respectively for specimen with optimum modulus at dry condition. A reduction in tensile modulus by 19.4% is noticed for 3rd sample immersed in distilled water. The possible reason may be due to increased water absorption results in fiber swelling providing resistance against deformation.

3.5 Flexural test

Fig. 7 shows the flexural strength of composite samples. Similar to tensile strength flexural strength values increase with increase in filler loading up to 5%. With further filler loading flexural strength reduced. A maximum flexural strength of 36.64Mpa for 5% filler is observed. An increase from 26.2Mpa for sample without filler to 36.64Mpa for sample with filler was observed. This due to improved distribution of matrix by the micro fillers allowing it penetrate through the piled up coir fibers. Reduction in flexural strength for 2nd sample observed is 28.23Mpa and 30.75Mpa for samples immersed in sea water and distilled water respectively. The reduction is flexural strength of samples immersed in distilled water for 1st, 2nd, 3rd and 4th specimen are 21.9%, 16.07%, 27.3% and 41.4% respectively. Specimens immersed in sea water showed a reduction by 28.16%, 22.95%, 36.7% and 56.59% for 1st, 2nd, 3rd and 4th specimen respectively. The value of reduction in flexural strength after immersion in sea and distilled water is less than that of tensile strength this is due to exhibition of less fiber pull out failure mechanisms when the specimen failed in bending test because in flexure, load applied is perpendicular to longitudinal direction of fiber reinforcement (Libo and Nawawi, 2015).

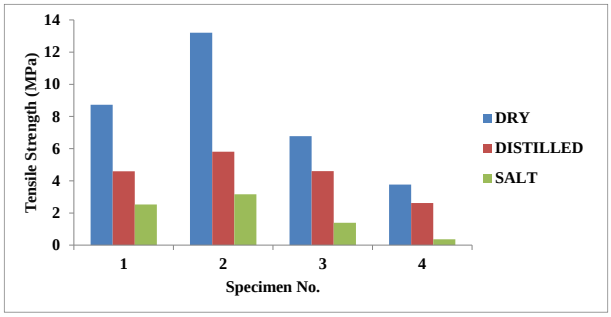

Fig. 5: Tensile strength of composites

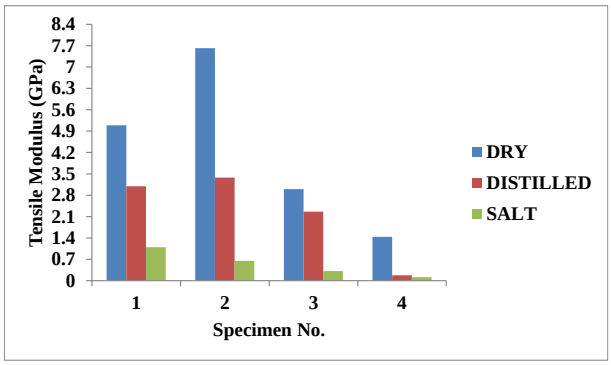

Fig. 6: Tensile modulus of composite

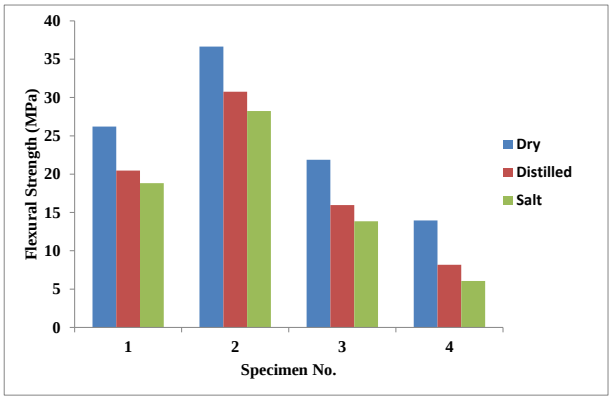

Fig. 7: Flexural strength graph

Flexural modulus in Fig. 8 defines resistance against stiffness of the material. Flexural strain of water immersed sample is higher than that of dry

ones. This could be due to high amount of water absorption causes swelling of fibers, which could fill up the gaps between fibers and polymer matrix eventually increasing the mechanical property (Karmaker et al., 1994). Flexural modulus is higher in sea water, because of salts and ions present fill up the gaps and cracks resulting in increased flexural modulus. Cellulose and integrity is lost when natural fiber composite tends to be ductile (Joseph et al., 2002). Here the water molecules act as plasticiser agent in composite material, which led to an increase of maximum strain of composites after absorption (Stamboulis et al., 2001). Flexural modulus of 1st and 2nd specimen immersed in distilled water showed an increase by 7.08% and 22.22% respectively. An increase by 55.11% and 25.9% for 1st and 2nd specimen immersed in sea water is observed. An increase in flexural modulus is due to fiber swelling covering up the gaps in specimen. Salt present in sea water covers up the smaller gap and voids showing higher resistance against deformation when compared to specimen (1st and 2nd) immersed in distilled water. 3rd specimen showed a reduction by 34.8% and 53.2% for distilled and sea water immersed specimens respectively. The possible reason may be higher amount of salt is settled due to higher void content which resulted in matrix-fiber debonding decreasing flexural modulus. Another possible reason is sufficient amount of salt may not have settled in the sample failing to clog the voids and pores resulting in decreased flexural modulus. 4th specimen immersed in distilled and sea water a reduction by 37.5% and 29.7% respectively.

3.6. Hardness test

Vickers Micro hardness test had conducted for the composites samples. Fig. 9 depicts that as the filler weight percentage increases, the hardness value of the composites increases. Maximum hardness of 33.3 is noticed with 4th sample. 44% increase is noticed in hardness value when compared to samples without filler material. This may be attributed to the fact that the incorporation of higher filler percentage into resin has lowered the mobility of the polymer chain in the rigid composites (Mir et al., 2013).

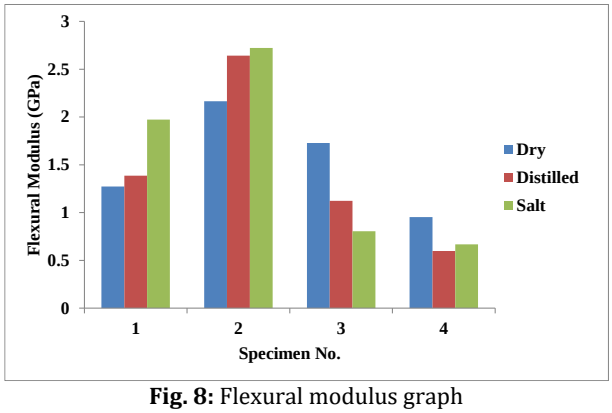

Fig. 8: Flexural modulus graph

Hardness increased with increase in filler loading as it led in improvement in matrix surface resistance to adhesion. However the hardness value is affected by water absorption. Hardness decreases in all coir fiber samples in wet condition and is associated with the poor interface between matrix and fiber by the water absorption. As the water absorption increases hardness value of composite decreases and found that deformation depth increased for water immersion specimen than dry ones. Due to water absorbing nature of fiber and filler, porosity of filler led to weak matrix fiber interface which was noticed in flax fiber reinforced composite (Dhakal et al., 2007). Moisture penetrating into polymer matrix through voids and cracks can break down the structure of matrix (Ashbee and Wyatt, 1969). When in dissolved state, NaCl exists in the sea water as cations and anions would penetrate along with the water molecule into composite, causing damage to matrix, fiber and their interface. Higher deterioration in Mechanical property is observed in sea water when compared to distilled water. The reduction in hardness value by 12.5%, 2.33%, 9.34%, 15.31% for 1st, 2nd, 3rd and 4th specimen immersed in distilled respectively in observed. Reduction by 18.45%, 3.31%, 16.63% and 23.31% is noticed in 1st, 2nd, 3rd and 4th specimen immersed in sea water respectively.

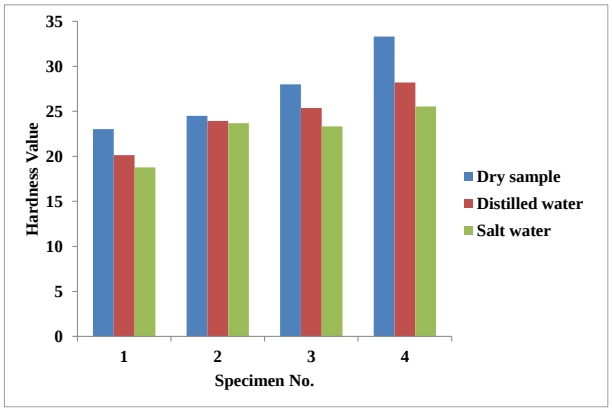

Fig. 9: Hardness characteristics graph

3.7. Morphological analysis

Figs. 10a-e shows the microscopic defects observed in composite specimen. Fig. 10a shows the cracking of matrix in composite sample with higher filler percentage subjected to water absorption. Due to insufficient and improper wetting by the matrix, Fiber swelling resulted in matrix cracking, thereby degrading the mechanical property (Hong, 2010). Fig. 10b shows the epoxy agglomeration at the surface of composite sample with no filler. Agglomeration is usually observed in long coir fibers as packing of fibers will not be efficient in the composite. This leads to regions which are high in matrix and thereby resulting in ease of failure at the bonding interfacial region (Pothan et al., 2003).

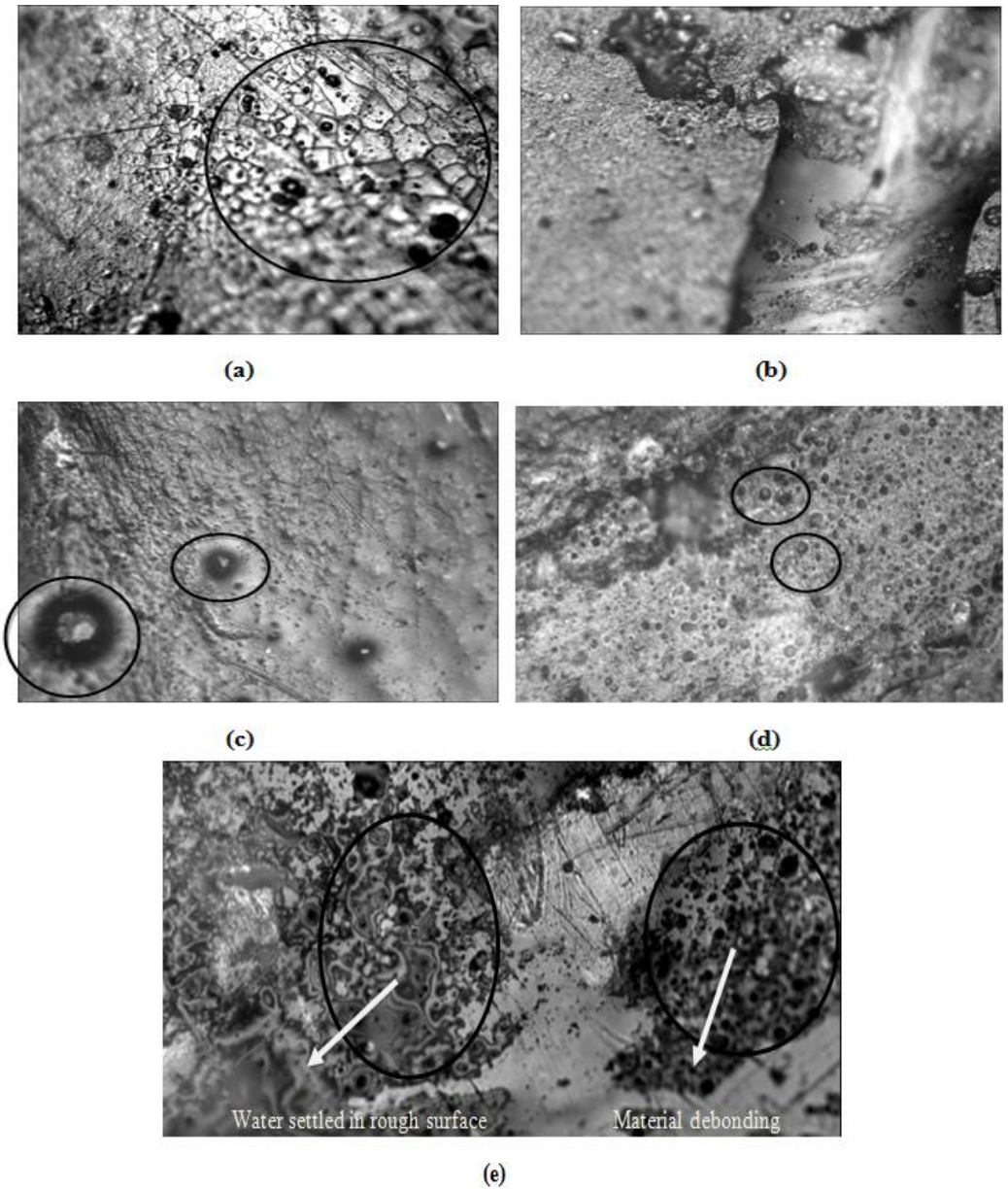

Fig. 10: Macroscopic defects in composite sample

Fig. 10c shows the sawdust agglomeration at higher filler percentage, which results in improper wetting of matrix due to the porosity of the sawdust, thereby reducing mechanical performance. Rough surface finishing is also observed in Fig. 10c which resulted in accumulation of salts present in sea water on the surface of the composite resulting in weight gain. Fig. 10d shows the pores formed due to exothermic heat produced during curing of matrix.

4. Conclusion

Tested samples showed that sawdust impregnation has improved the mechanical properties of long coir fiber epoxy composite up to certain percentage. As the filler percentage increased due to the reduction in epoxy percentage and curly nature of fiber led to non-uniform fiber-matrix bonding which resulted in failure. Main reasons for failures are agglomeration of materials, piled up coir fibers due to its length, curly nature of fibers and traditional fabrication and fiber treatment method.

List of symbols

σ_t	Tensile strength
E_t	Tensile Modulus of elasticity
σ_f	Flexural Strength
E_f	Flexural Modulus
D	Diffusion Coefficient
$W2$	Final weight of specimen
$W1$	Initial weight of specimen
B	Thickness of specimen immersed in water
M_s	Saturation moisture percentage
L	Span length of the sample
P	Maximum load
b	Width of specimen
t	Thickness of specimen
d	Displacement

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