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Effect of surface treatment on hair fiber as reinforcement of HDPE composites: Mechanical properties and water absorption kinetics

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Abstract−The objective of this work was to investigate the effect of surface improvement on human hair fiber (HF) based high-density polyethylene (HDPE) reinforced polymer composites. A universal testing machine was used for the characterization of the mechanical behaviors of different types of HF base reinforced HDPE composites, and studies were conducted for the optimization of male/female fiber percentage (15% HF) in polymer composites. The alkali treatment (AT) and acrylic acid treatment (AAT) of HF reinforced HDPE composites showed a remarkable improvement in tensile strength (upto 15.487 MPa and 15.638 MPa, respectively), which was significantly changed in comparison to the tensile strength of untreated composites. FTIR and SEM test were used to characterize the fiber surface and HF/HDPE reinforced composites. Water absorption kinetics was investigated for the study of diffusion mechanism and kinetics of composites materials, which would be useful to boost the applications of the composite in different areas.

Keywords: High Density Polyethylene, Composite, Chemical Treatment, Mechanical Behavior, Water Absorption Kinetics

INTRODUCTION

Present era shows the importance of natural fiber based polymer reinforced composites due to environmental and economic purposes. More efficient and eco-friendly polymer-based composites are developed with natural fibers (plant and animal hairs). However, the adhesion property between fiber and matrix is decreased due to fiber irregularity (size, variable quality, water sensitivity, etc.). Therefore, different types of fiber treatment are performed to enhance the adhesion property. Hair fiber (HF) comes from natural fibers (animal fibers) which have some attributes as good strength, eco-friendly and lightweight. Most of society (rural and urban) considers hair fiber as a waste material easily found in municipal waste streams [1]. Keratin (protein) is a major constituent of hair fiber, which has 65-95% of proteins, and the rest is water and lipid pigments. Cuticle, cortex, and medulla are three parts of the hair fiber. The cortex (keratin) is responsible for the mechanical property [2]. The long chains of the cortex (keratin) are compressed to form a regular structure which provides the strength in hair fibers [3]. High keratin content corresponds to rich disulfide cross-links, leading to high mechanical properties. The average chemical compositions of Indian hair for sulfur, nitrogen, carbon, hydrogen and oxygen are 4.82, 15.40, 44.06, 6.53 and 29.19%, respectively [4]. Hair fiber has good tensile strength and is also readily available in municipal solid waste. Hair is a low-density natural fiber, which can be used for the development of natural fiber based reinforced polymer composites. The uses of hair fiber reduce the waste stream and also contribute to the economy. Usually, the hair diameter ranges between 50-80 μm, density around 1.32 g/cm^3, tensile strength ranges 150-350 MPa and elongation at break 20-40% approximately [3,5].

The HF/HDPE-reinforced composite has the advantage that it is completely biodegradable and renewable [6,7]. Different types of accessories (dash boards, railings, door frames and panels, mobile and laptop cases, etc.) can be developed through hair fiber reinforced HDPE composites due to good mechanical properties and high corrosion resistance properties [8,9]. Adhesion between matrix-fiber is the major aspect of improving the strength of reinforced composites. The additional reactive sites on the fiber surface are developed by surface treatment of fiber, which enhances the bonding between fiber-matrix. Indeed, several authors characterized it as the mechanical behavior and water absorption properties of natural fiber reinforced polymer composites [10-13]. Srivastava and Sinha [14] studied the different mechanical and water absorption properties of alkali treated HF/HDPE reinforced polymer composites. Mittal et al. [15] did a literature study of different fiber/polymer composites and also compared the properties of reinforced composites. Mittal and Sinha [16,17] investigated the mechanical and water absorption properties of bagasse fiber-reinforced epoxy composites and wheat straw fiber-reinforced epoxy composites. They also optimized the fiber loading ratio in reinforced composites for achieving the optimum properties. Imoisili et al. [18] studied the water absorption behavior of cocoa-pod epoxy composites and estimated the diffusion mechanism/kinetics from experimental data. Ndapeu et al. [19] estimated the water absorption kinetics with the help of experimental data of coconut shells. Srivastava and Sinha [20] investigated the structural and thermal properties of alkali treated HF/HDPE reinforced polymer composites.

The aforementioned researchers developed natural fiber based

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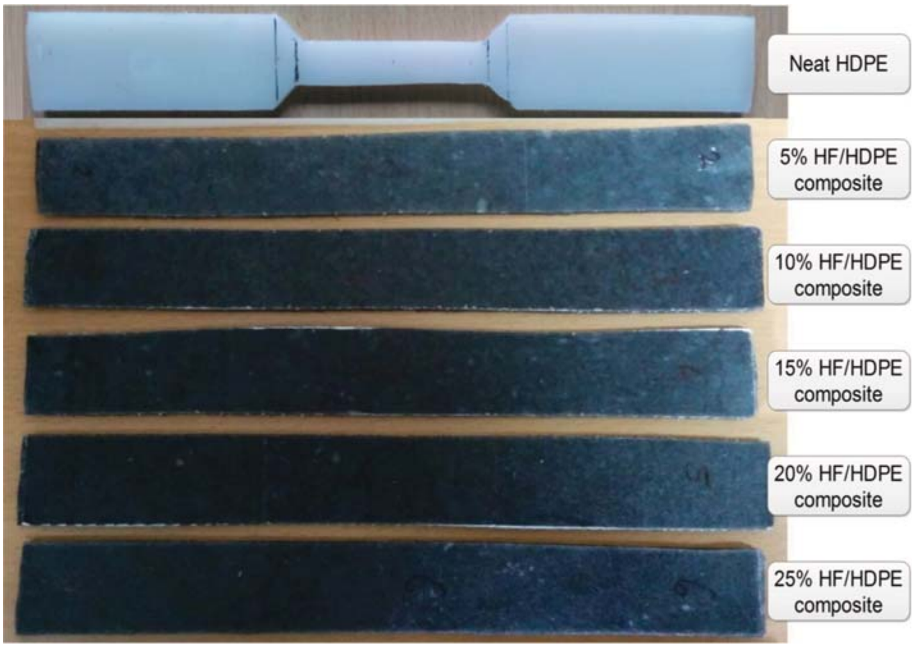


Fig. 1. Specimens prepared as per ASTM standards.

reinforced polymer composites and studied the different mechanical behavior and water absorption properties which are improved with the help of fiber treatment and the coupling agent. In the present work, we deal with various types of hair fibers based reinforcement HDPE composites and have studied the effect of hair fiber treatments on mechanical behavior and water absorption kinetics of HF/HDPE reinforced with a different range of fiber loading (5, 10, 15, 20 and 25 wt%).

MATERIALS AND CHARACTERIZATION

1. Material and Preparation of Composites

Waste human hair fiber of both male and female within the age group of 18-23, 24-29 and mixed age (Mixed age: random age HF) was purchased from the local parlor in Roorkee, India. The fiber length and thickness were about 5-10 mm and 50-80 μm, respectively. Neat HDPE natural powder was purchased from Rapid Coat Powder Coatings Pvt. Ltd. Ghaziabad, India. HDPE has excellent mechanical strength, toughness, resistant to the moisture and chemical attack and superior bonding nature. Sodium hydroxide (assay-min 98.0%) and acrylic acid (assay-min 99.0%) were procured from Leonid Chemicals Pvt. Ltd. Bengaluru, India. Ethanol absolute (assay-min 96.4%) was purchased from Taj Pharmaceuticals Ltd. Gujarat, India [14].

Waste HF was used directly as a fibrous material after different treatments. The hair fiber was washed with hot water to remove its oil content and other adhered impurities followed by treatment with absolute ethanol for the removal of organic components adhering to HF [6]. This HF sample was washed several times with distilled water to remove alcohol residues, and was dried in a hot oven for 48 hr to remove the water content. Alkali treatments (AT) and acrylic acid treatments (AAT) were used for the surface treatment of different groups of human HF (male/female). HF was soaked with 0.25 N and 0.5 N NaOH at room temperature for 1 hr, and followed by 0.07 N, 0.14 N and 0.21 N acrylic acid for 1 hr at room temperature [14]. Again, these fibers were washed several times with distilled water to remove unwanted adhered impurities, followed by drying in a hot air oven at 70 °C for 48 hr. During these treatments, a liquor ratio of 20 : 1 was maintained throughout [7,14].

Hot compression molding technique was used for developing the hair based natural fiber reinforced HDPE composites. A different range of hair fibers (male/female) was used with a specific length and mixed with matrix phase uniformly by stirring for 10-15 min at 1,800 rpm. A homogeneous mixture of HF and neat HDPE was obtained, which varied in the different ratios according to the fiber loading (5, 10, 15, 20 and 25 wt%) in composites. The homogeneous sample was fed into the mold and the temperature was raised to 180 °C of molding machine with specific dead load 25-30 kg. After removal of mold from the molding machine, the temperature of the mold was decreased naturally up to 120 °C. The dimension of the prepared composite was $300\times300\times4$ mm^3. The mold was coated with a Teflon sheet to avoid sticking problem of polymer composites. The specimens of the composite were prepared according to the ASTM standards, which are shown in Fig. 1 [7,14,19].

CHARACTERIZATION

1. Mechanical Properties Test

The mechanical properties of flat polymer specimens were investigated by using the computerized universal testing machine (UTM, INSTRON model 5982). The specimens were prepared for tensile and flexural analysis as per ASTM D3039 and ASTM D790, respectively. A constant crosshead speed of 2 mm/min was applied during the material testing for better observation. The values of the analyses were taken by the average of four same types of samples for each composition [11-16].

2. Fourier Transformation Infrared Spectroscopy (FTIR)

FTIR spectrometer (Nicolet 6700 series, Nottingham UK) was used for the investigation of untreated and treated HF/HDPE reinforced composites. Potassium bromide (KBr) as a reference component was used for preparing the pellets of testing materials. Spectrum resolution of 4 cm^{-1} was used for analysis of samples with range 4,000-600 cm^{-1} [17-20].

3. Scanning Electron Microscopy (SEM) Analysis

SEM (Model LEO-435 VP, USA) was used to assess the morphology of untreated and treated hair fiber as well as reinforced composites with an acceleration voltage of 0-30 KV. Fractured surfaces of the polymer composite were used for the study of the failure mode of composites after the mechanical test. A thin layer coating of gold is used for SEM analysis of fractured surfaces [14,20].

4. Kinetics of Water Absorption

ASTM D570 standard was used for moisture absorption test of composite samples. High precision balance (Voyager Analytical Balances CPA225D, USA) was used to estimate the gain weight difference for water absorption test of the samples. The % gain of water absorption was calculated by the following formula:

$$\text{Water absorption (\%)} = \frac{W_t - W_i}{W_i} \times 100$$

where W_t is measured weight of the specimen at time interval and W_i is the initial dry weight of the specimen.

Diffusion behavior in glassy polymers can be classified according to the relative mobility of the penetrant and of the polymer segments. Fick's theory was used for the study of water absorption kinetics and diffusion mechanism of HF/HDPE reinforced polymer composites [18,19]. Water absorption kinetics can be distinguished by the shape of the sorption curve represented by the fol- lowing equations:

$$\frac{M_t}{M_s} = Kt^n \qquad (1)$$
$$\log\left(\frac{M_t}{M_s}\right) = \log K + n\log t$$

where, M_t is the moisture content at time t; M_s is the maximum moisture content at the equilibrium; and k and n are constants.

Fick's theory can predict the water absorption processes of natural fibers and fillers reinforced polymer composite. Water absorption rate linearly decreases with square root of time up to equilibrium plateau [18]. The diffusion coefficient (D) can be determined from the following equation:

$$\frac{M_t}{M_s} = \left(\frac{4}{h}\right) \times \left(\frac{D}{\pi}\right)^{1/2} \times (t^{1/2}) \qquad (2)$$

where, h is the thickness of sample.

RESULTS AND DISCUSSION

1. Mechanical Properties of HF/HDPE Reinforced Composites

Investigation of mechanical properties of HF/HDPE reinforced

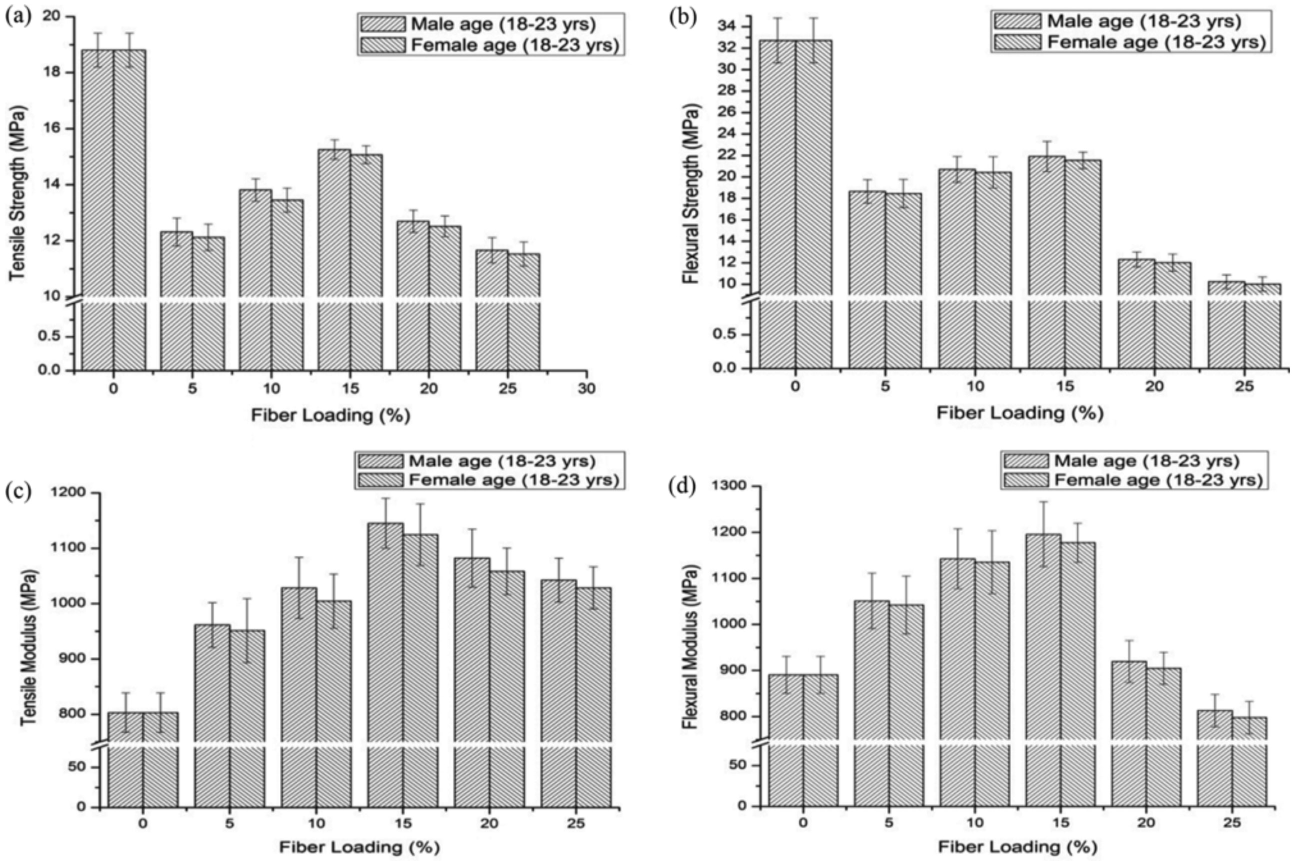


Fig. 2. Male/female HF/HDPE composites: (a) Tensile strength; (b) flexural strength; (c) tensile modulus; (d) flexural modulus.

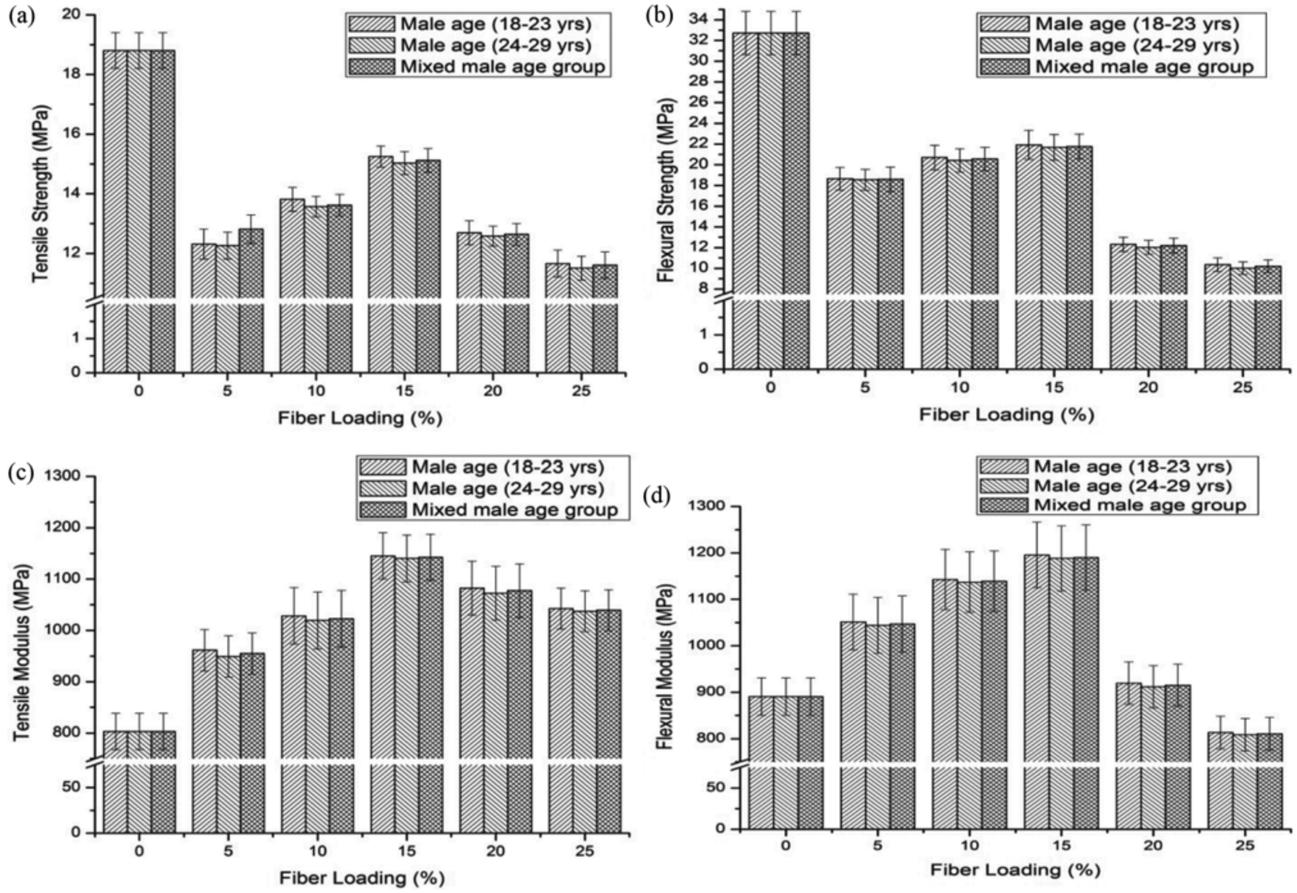


Fig. 3. Untreated male HF/HDPE composites: (a) Tensile strength; (b) flexural strength; (c) tensile modulus; (d) flexural modulus.

polymer composites is shown in Figs. 2-5. Mechanical properties of the polymer composites were affected with fiber types (male/female), age groups of fibers and the fiber loading (5, 10, 15, 20 and 25 wt%) in the composite mixture. The fiber pullout, voids, poor interlocking, and non-uniform stress transfer are responsible for the decrement in mechanical properties [12-16].

Mechanical properties (tensile and flexural) of the untreated male/female HF based reinforced HDPE composites are shown in Figs. 2(a)-(d). In this investigation, tensile strength and its modulus increased with increasing the loading up to 15%, and thereafter, the properties decreased with the percentage of fiber in polymer increases due to poor bonding between fiber/matrixes. A similar pattern was observed in a flexural strength case which increased up to 15% fiber loading in polymer, and above this percentage of fiber loading these properties were also decreased. Tensile and flexural strength were reached up to 15.254 MPa and 21.914 MPa which was less than neat HDPE matrix. But, the tensile and flexural modulus were found to be 42.63% and 34.22%, which was more than neat HDPE matrix. At 15% fiber loading, the male fiber based HF/HDPE reinforced composites was found with improved tensile strength by 1.22% and flexural strength by 1.73% as compared to female based HF/HDPE reinforced composites. A similar pattern was achieved for tensile modulus and flexural modulus, which was 1.52% and 1.83% more in comparison to female based 15% HF/HDPE reinforced composite. The mechanical behavior of the composites depended on the properties of the fibers (male/female), which were affected by size and diameter of the fibers. Male hair has high rich disulfide cross-links as compared to female hair, which leads to good mechanical properties [6].

Tensile and flexural behavior of untreated male hair fiber (18-23, 24-29 and mixed age groups) based reinforced HDPE composites are shown in Figs. 3(a)-(d). The age groups of the male HF played a major role in the investigation of mechanical properties of HF/HDPE reinforced composites. The male age group 18-23 years was shown to have optimum mechanical strength in comparison to age group 24-29 years and mixed age group of HF. Mechanical behavior of the composites depended upon the fiber strength, bonding nature, fiber quality, and fiber geometry. This concluded that the strength of the hair fiber depends upon the age of the persons and uses of different chemicals products. Reinforced composite of the age group 18-23 years HF depicted superior mechanical results due to their healthy nature of the fibers.

Mechanical behavior of male (age 18-23 years) untreated and treated HF based reinforced HDPE composites is shown in Figs. 4 and 5. The characterization of applied load and their extension of untreated and chemical treated hair fiber based HF/HDPE reinforced composites are shown in Figs. 4(a)-(b). The load behavior of the composites depended upon the adhesion between matrix-fibers, which was observed to be maximum at 15 wt% fiber loading in composites. Load bearing strength of composites was slightly

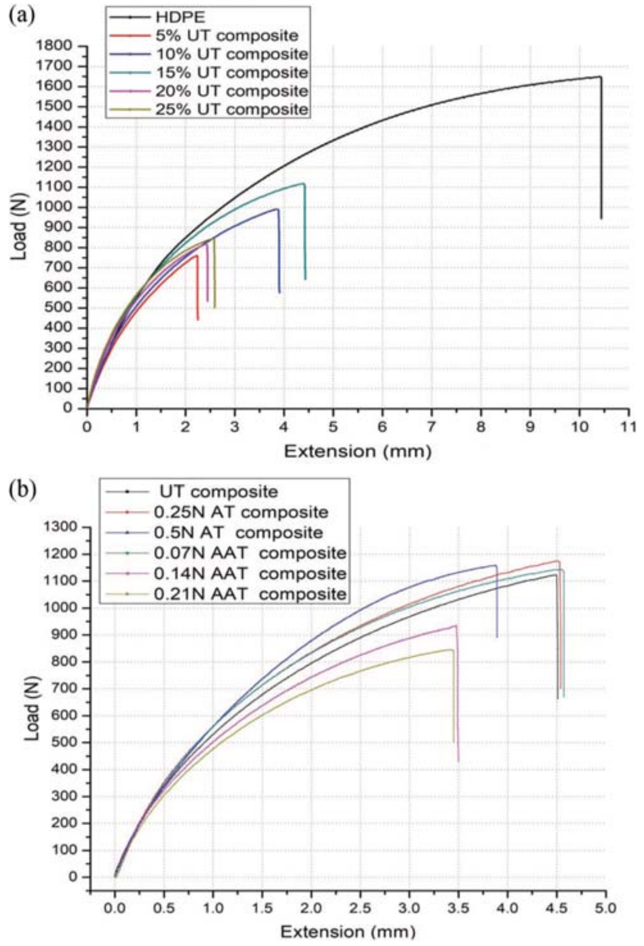


Fig. 4. (a) Effect of fiber loading on Load vs Extension of untreated HF/HDPE reinforced composites; (b) effect of different modifications techniques on load vs extension of HF/HDPE composites with 15 wt% hair fiber (UT: Untreated; AT: Alkali treated; AAT: Acrylic acid treated).

changed with surface modification of fibers. Optimum load strength was investigated with 0.07 N AAT HF/HDPE reinforced composites. Superior strength was observed at 15% fiber loading in the polymer, which can be useful to reduce the cost of material and the utilization of the waste hair fiber from the environment.

Tensile and flexural behavior of treated and untreated HF based reinforced HDPE composites are shown in Figs. 5(a)-(b). The surface modification (Alkali and acrylic treatment) of HF was also affected by tensile and flexural properties of reinforced polymer composites. 0.25 N and 0.5 N alkali treatments were applied on the HF surface, which slightly affected the surface of HF in comparison to untreated HF. The strength of the fiber was increased at 0.25 N alkalizing, and a further 0.5 N alkali treatment damaged the fiber surface as shown in SEM images. Mechanical results showed that 0.25 N alkali treatment was the optimum treatment ratio for HF/HDPE reinforced composites. Thereafter, an acrylic treatment (0.07 N, 0.14 N and 0.21 N AAT) applied on hair fiber followed by 0.25 N alkali treatment. 0.07 N AAT was an optimum range of treatment for maximum strength of fiber based reinforced composites as shown in SEM. Furthermore, 0.14 N and 0.21 N AAT of

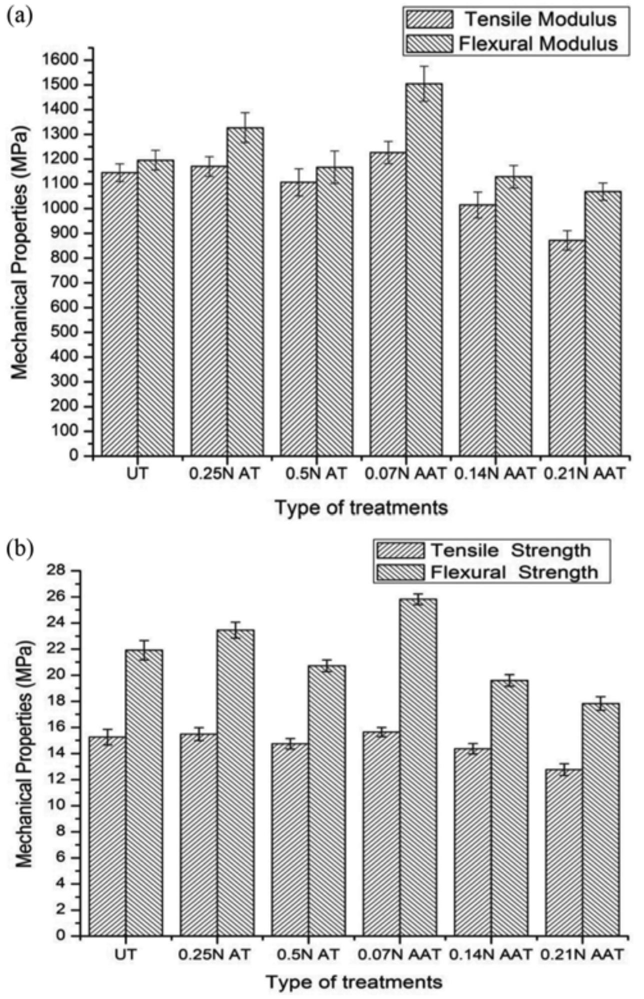


Fig. 5. (a) Effect of different modifications techniques on the tensile and flexural strength of HF/HDPE composites with 15 wt% hair; (b): Effect of different modifications techniques on the tensile and flexural modulus of HF/HDPE composites with 15 wt% hair fiber (UT: Untreated; AT: Alkali treated; AAT: Acrylic acid treated).

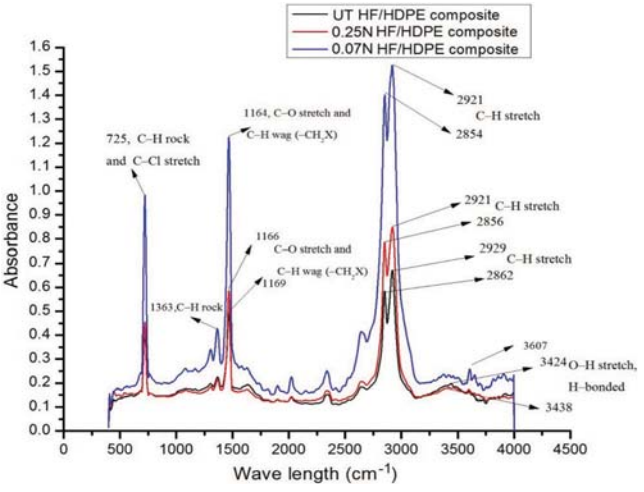


Fig. 6. FTIR spectra of the treated and untreated HF/HDPE reinforced composites.

fiber created more roughness on the fiber surface, which damaged the strength of the fibers. Alkali and acrylic treatment techniques enhanced it up to 15.487 MPa and 15.638 MPa, respectively, which provided more strength as compared to untreated fiber based reinforced composites at 15% fiber loading. A similar pattern was obtained in flexural strength cases which were 23.456 MPa and 25.818 MPa, respectively. Alkali and acrylic treatment of fibers enhanced tensile modulus up to 45.77% and 52.79%, which was greater than the neat matrix. A similar pattern was observed in flexural modulus which was 48.98% and 68.92% more efficient in comparison to the neat polymer.

The aforementioned results of HF/HDPE reinforced composites depicted that the optimum results were observed in the different types of composites (15%UT HF/HDPE, 0.25 N AT 15% HF/

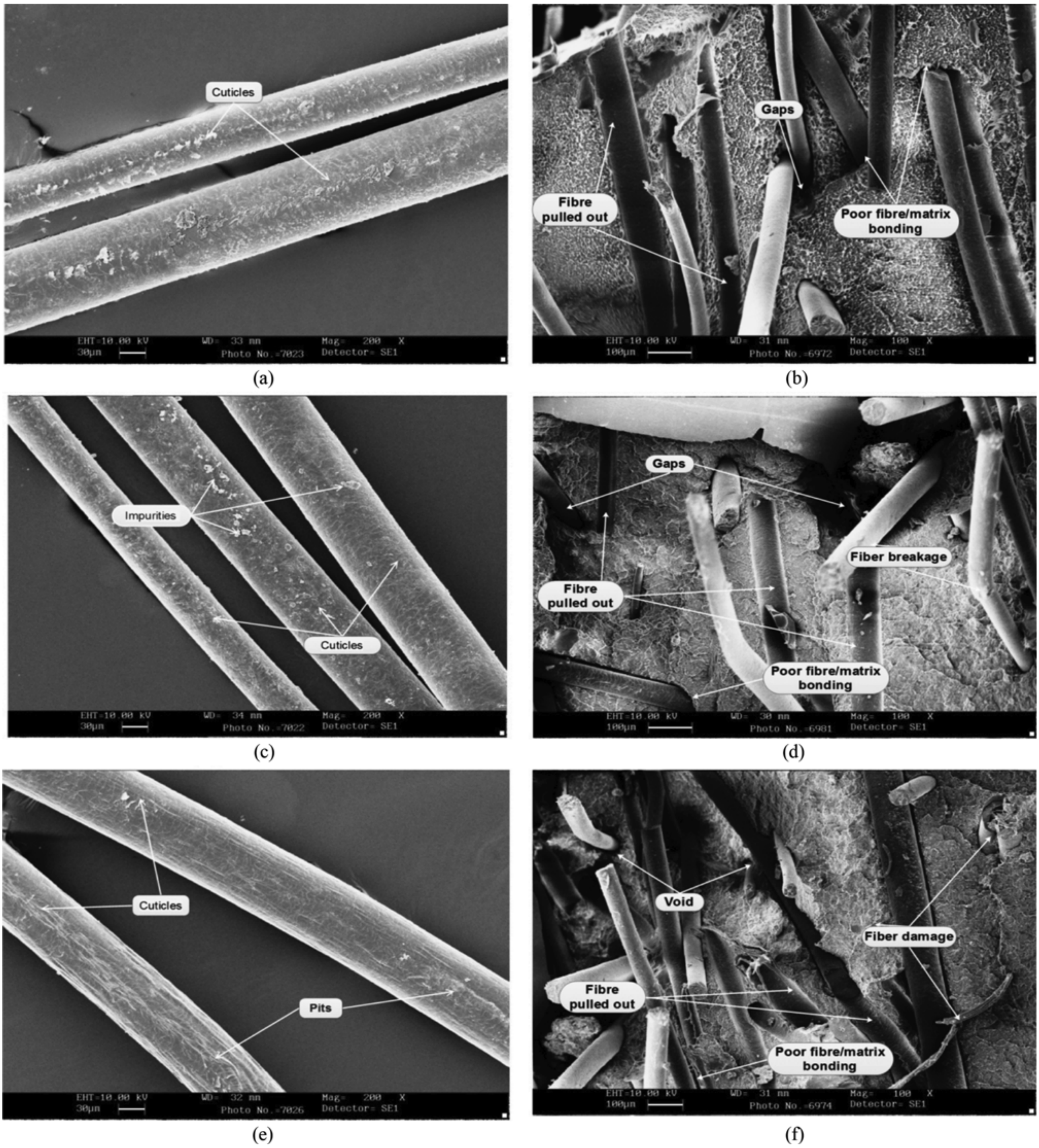


Fig. 7. SEM micrographs: (a) UT fiber; (b) 0.25 N AT hair fiber; (c) 0.07 N AAT hair fiber; (d) UT 15% HF/HDPE reinforced composite; (e) 0.25 N AT 15% HF/HDPE reinforced composite; (f) 0.07 N AAT 15% HF/HDPE reinforced composite (UT: Untreated; AT: Alkali treated; AAT: Acrylic acid treated).

HDPE and 0.07 N AAT 15% HF/HDPE composites). And, the maximum mechanical stability of composites was observed at 0.07 N AAT 15% HF/HDPE reinforced composites.

2. FTIR Analysis of HF/HDPE Reinforced Composites

FTIR spectra of untreated (UT) and treated (alkali and acrylic acid) human hair fiber based reinforced HDPE composites are shown in Fig. 6. Optimum results of mechanical properties were found at 15% HF loading in composites with 0.25 N alkali treatment (AT) and 0.07 N acrylic acid treatments (AAT). According to this study, the absorbance rate was improved with alkali treatment up to 0.25 N, and beyond the above treatment, the property was going down. A similar pattern was observed in acrylic acid treatment, which was optimal at 0.07 N AAT and after that, the fiber strength tended to decrease. The FTIR spectra showed a broad peak of 0.07 N AAT fiber composite at 725 cm^{-1} corresponding to the C-H rock and C-Cl stretching vibration group. One more peak of all three types of fiber composites occurred at around 1,164-1,169 cm^{-1}, which represented the C-O stretching vibration and C-H wag (-CH_2X) groups. Another two broad peaks occurred in all three types of fiber composites between 2,854-2,929 cm^{-1}, which represented the C-H stretching vibrations. Many other peaks of UT, 0.25 N AT and 0.07 N ATT fiber composites were observed between 3,424, 3,438 and 3,607 cm^{-1}, which represented the O-H stretching vibration and H-bonded group. These changes were found due to the moisture present in composites [7,14,20].

3. Scanning Electron Microscopy (SEM) Analysis of HF/HDPE Reinforced Composites

SEM was used for the study of UT, AT and AAT hair fiber as well as their reinforced HF/HDPE composites which are shown in Figs. 7(a)-(f). Amino acids and cuticles were presented in HF, which was responsible for smooth surface and weak bonding between fiber/matrix. Untreated HF and HF/HDPE composites are shown in Figs. 7(a) and 7(d). But, the surface modification (alkali/acrylic) was the key factor to enhance the bonding strength of fibers. 0.25 N alkali treatment was done to remove the impurities and was responsible for the formation of new cuticles on the fiber surface, which enhanced the bonding strength of composites. 0.25 N AT hair fiber and their composite are shown in Fig. 7(b) and 7(e), respectively. A similar pattern was observed in the acrylic acid treatment of fibers. 0.07 N AAT removed more impurities and thus more roughness was formed on the fiber surface, which is shown in Fig. 7(c) and their composite surface is shown in Fig. 7(f). AT and AAT of HF increased the pit on fiber surface, which improved the mechanical behavior of composites and developed the next generation reinforced HF/HDPE polymer composites [14].

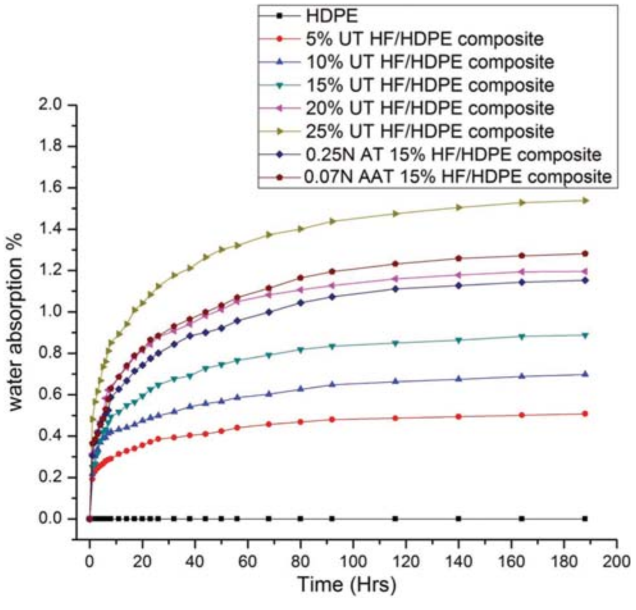


Fig. 8. Water absorption behavior of HF/HDPE composites (UT: Untreated; AT: Alkali treated; AAT: Acrylic acid treated).

4. Water Absorption Kinetics

The water absorption behavior of untreated and treated HF/HDPE composites is shown in Fig. 8. It is clear from the figure that absorption of water increased with an increase in the fibers loading in composites. Water absorption of composites depended upon the fibers surface and bonding between matrix-fibers. In HF/HDPE composites, absorption percentage was less in comparison to other natural fibers due to hydrophobic nature. The % gain of water absorption was increased after applying the 0.25 N alkali treatment of 15% HF/HDPE composite, because alkali treatment increased the pits/roughness on fiber surface as shown in SEM images. But 0.07 N acrylic acid treatment was responsible for the improvement of water absorption due to optimum bonding between fiber/matrix. SEM images showed the morphology of reinforced composites, which was helpful for the observation of water sorption test [14,15].

Fick's theory is used to investigate the kinetics and diffusion mechanism of reinforced HF/HDPE composites [17,18]. Kinetics parameters are estimated by putting the experimental values in the following equations:

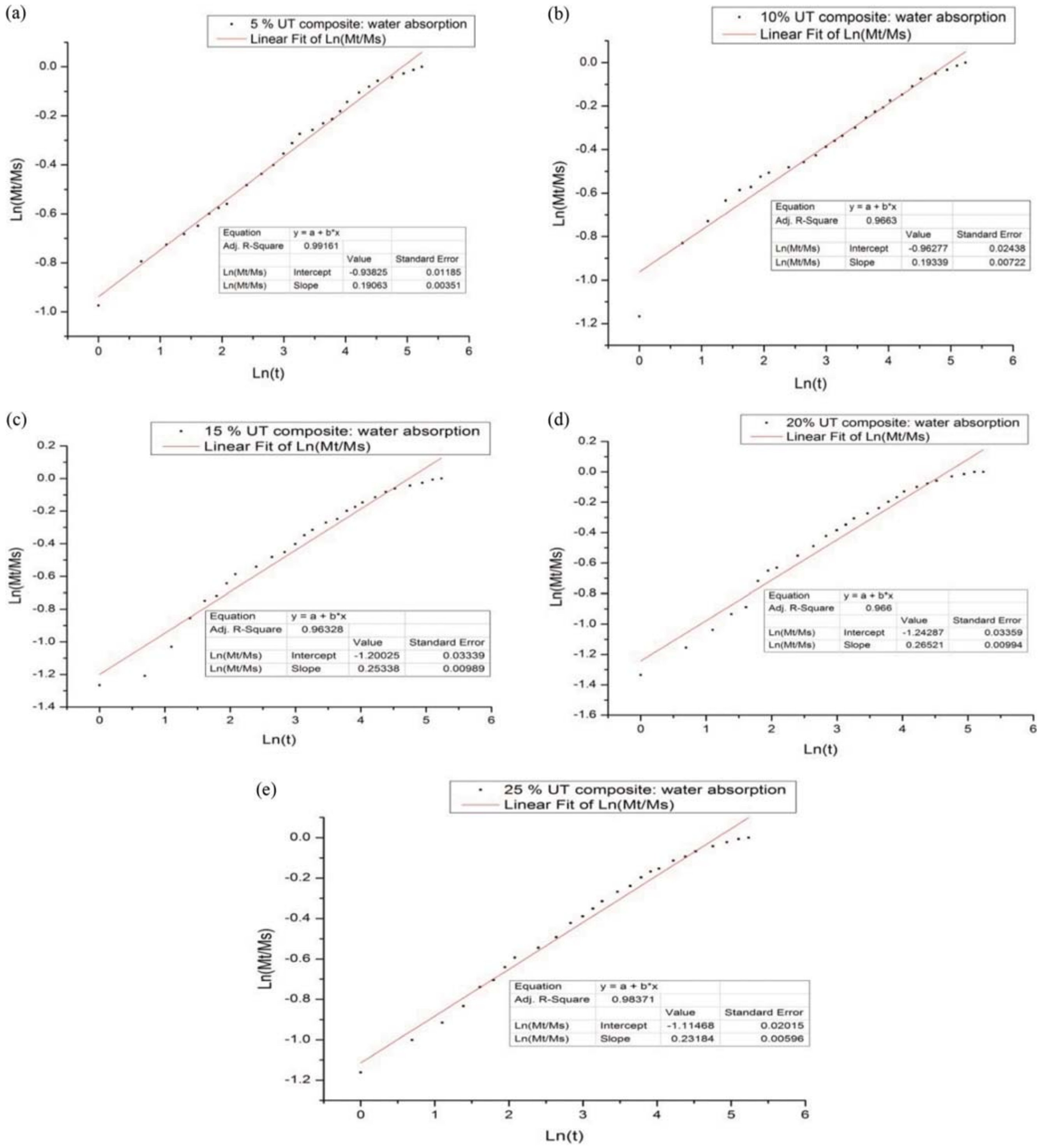
$$\log\left(\frac{M_t}{M_s}\right) = \log K + n\log t \tag{1}$$

$$\frac{M_t}{M_s} = \left(\frac{4}{h}\right) \times \left(\frac{D}{\pi}\right)^{1/2} \times (t^{1/2}) \tag{2}$$

Figs. 9(a)-(e) show the curve fitting of the experimental values of water absorption test of untreated composites in Eq. (1), and Figs. 10(a)-(c) show the curve fitting of the experimental values of water absorption test of 15% HF/HDPE reinforced composites in Eq. (1). The optimum values of parameters (n and k) were estimated via curve fitting method, which was helpful for the estimation of the mechanism. The initial diffusion coefficient of each composite was calculated by Eq. (2), which provided the information of water absorption pattern of composites. Kinetics and diffusion mechanism of HF/HDPE reinforced composites are shown in Table 1. 0.07 N AAT 15% HF/HDPE composite showed the optimum results of diffusion which was least diffused in comparison to other composite materials. The study of water absorption kinetics was used for the development of advanced and water resistant composites [19].

CONCLUSION

Various mechanical properties of male/female hair fibers were



(a)

Equation	y = a + b*x		
Adj. R-Square	0.99161		
		Value	Standard Error
Ln(Mt/Ms)	Intercept	-0.93825	0.01185
Ln(Mt/Ms)	Slope	0.19063	0.00351

(b)

Equation	y = a + b*x		
Adj. R-Square	0.9663		
		Value	Standard Error
Ln(Mt/Ms)	Intercept	-0.96277	0.02438
Ln(Mt/Ms)	Slope	0.19339	0.00722

(c)

Equation	y = a + b*x		
Adj. R-Square	0.96328		
		Value	Standard Error
Ln(Mt/Ms)	Intercept	-1.20025	0.03339
Ln(Mt/Ms)	Slope	0.25338	0.00989

(d)

Equation	y = a + b*x		
Adj. R-Square	0.966		
		Value	Standard Error
Ln(Mt/Ms)	Intercept	-1.24287	0.03359
Ln(Mt/Ms)	Slope	0.26521	0.00994

(e)

Equation	y = a + b*x		
Adj. R-Square	0.98371		
		Value	Standard Error
Ln(Mt/Ms)	Intercept	-1.11468	0.02015
Ln(Mt/Ms)	Slope	0.23184	0.00596

Fig. 9. Diffusion curve fitting plot of untreated HF/HDPE composites, (a) 5%UT; (b) 10%UT; (c) 15%UT; (d) 20%UT; (e) 25%UT.

compared in which superior composites of male hair fiber were also compared with a range of age groups. The effect of male hair fibers with chemical treatment was investigated in terms of mechanical strength and modulus. Different chemical treatments (AT and AAT) of fiber were applied for enhancing the strength of composites. The experimental results showed that tensile and flexural properties of HF/HDPE reinforced composites were increased with the increase in the fiber loading upto 15 wt%, and beyond the 15 wt% of fibers, mechanical properties were relatively decreased. The optimum mechanical strength was found in 0.07 N AAT, which was followed by 0.25 N AT. Further, an excess AAT decreased composite strength due to damage of fiber surface. Alkali and acrylic treatment techniques enhanced the tensile strength, up to 15.487 MPa and 15.638 MPa, respectively. The treatment techniques provided

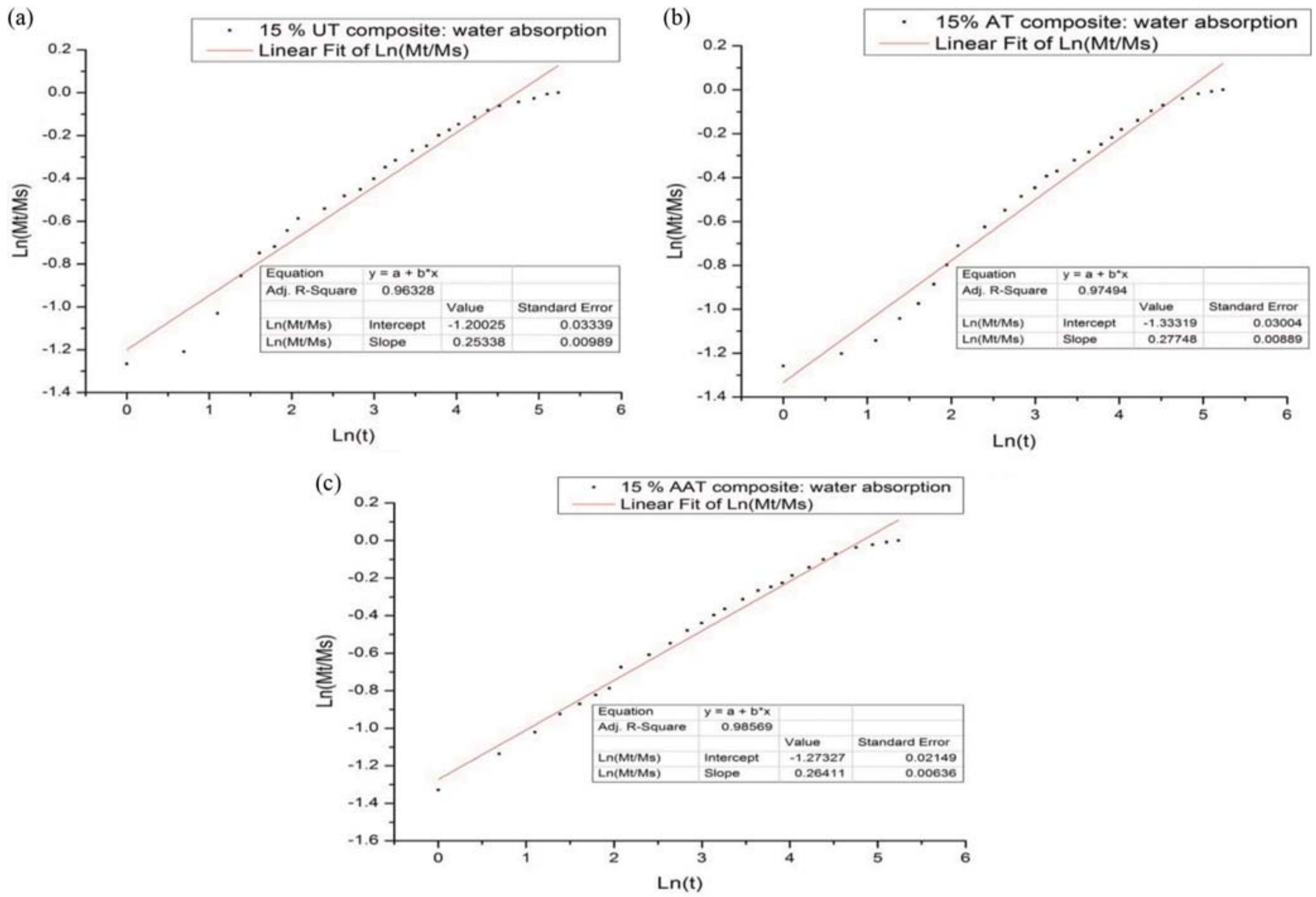


Fig. 10. Diffusion curve fitting plot of untreated and treated HF/HDPE composites at 15% fiber composition, (a) UT 15% composite; (b) 0.25 N AT 15% composite; (c) 0.07 N AAT 15% composite.

Table 1. Water absorption constant and diffusion coefficient of HF/HDPE composites

Sample	n	k	Diffusion $D*10^{-6}$ (m^2/s)
5% UT HF/HDPE composite	0.19063	0.391312	66.7014
10% UT HF/HDPE composite	0.19339	0.381834	55.0023
15% UT HF/HDPE composite	0.25338	0.301119	49.8268
20% UT HF/HDPE composite	0.26521	0.288555	46.9318
25% UT HF/HDPE composite	0.23184	0.32802	55.3065
1% AT 15%HF/HDPE composite	0.27748	0.263635	50.193
0.5% AAT 15% HF/HDPE composite	0.26411	0.279915	46.7191

more strength in comparison to untreated fiber based HF/HDPE reinforced composites. AT and AAT increased the pit on fiber surface, which improved the mechanical behavior of composites and developed the next generation reinforced HF/HDPE polymer composites. The effects of surface modification of fiber and their composites were investigated in terms of interlocking and morphology between the HF/HDPE. The % gain of water absorption was increased with the application of alkali treatment of fiber, because alkali treatment increases the pits/roughness on the fiber surface. 0.07 N AAT 15% HF/HDPE composite showed the optimum results of diffusion coefficient, which found the least diffused as compared to the other composite materials. Waste hair fiber can be replaced with other fiber for the development of new polymer composite materials, which provided good strength and environmentally friendly substances.

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