Heat and Chemical Treatments for Sawdust/UPE Composites

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ABSTRACT
Sawdust filler taken from White Cham (a type of wood) were mixed with unsaturated polyester (UPE) at 20, 30 and 40% volume fractions. the mechanical and physical properties of the composites treated by NaOH alkali at 0.5, 1, 1.5 and 2% (W/V) along the effect of volume fractions were investigated .The results showed improving in properties comparing with untreated sawdust filler, while heat/alkali treatment at 2%NaOH at different temperature 40, 60, 80 and 100 ˚C also showed improving in properties .the optimal mechanical properties had been obtained at 1% NaOH, and detracted at 2% NaOH. Yet, the mechanical properties at 2% NaOH were improved when heat treated. The optimal mechanical properties were obtained at 80 ˚C. It is worth mentioning that the optimization process was based on the tensile test. Also the thermal conductivity results show better insulation after the treatments especially at 100 ^{o}C.
Keywords: Unsaturated polyester, Sawdust, Mechanical and physical properties.

INTRODUCTION
Recently, composite materials have successfully substituted the traditional materials in several light weight and high strength applications. The reasons why composite are selected for such applications are mainly their high strength to weight ratio, high tensile strength at elevated temperatures, high creep resistance and high toughness [1]. The major fibers in use today are glass, Carbon and aramid. Recently research on engineering interest have been shifting from traditional synthetic fiber composite due to their advantages like high strength to weight ratio, non-carcinogenic and biodegradability. The term natural fiber covers abroad range of vegetables, animal and mineral fibers. Availability of natural fibers and easy of manufacturing is tempting researcher to try locally available inexpensive natural fibers as reinforcement material fibers as reinforcement material in polymer matrix [2]. The major disadvantages of natural fiber reinforced composites (NFRPCs) are the incompatibility between the hydrophilic natural fiber and hydrophobic polymer leading to formation of narrow and weak interphase. This could also lead to the non-uniform dispersion of fiber with the matrix. Interphase formed in (NFRPC) are relatively weak compared to conventional composite made of glass, Carbon or aramid due to the inherent polar and non-

polar nature of fiber and polymer respectively. This can be improved only by either physical or chemical modification of the fiber or polymer. Various surface modification have been developed in order to improve the composite,(i.e.) ; the natural fiber made less hydrophilic [3]. Alkaline treatment or mercerization is one of the best used chemical treatments for natural fibers [4]. Due to alkali treatment there is an increase in the amount of amorphous cellulose at the expense of crystalline cellulose. By this treatment there is a removal of hydrogen bonding in the network structure. Reaction which takes place during this treatment is shown below:

$\mathbf{NF - OH^{+} + Alkaline\ treatment\ (NaOH) \dashrightarrow NF - O - Na + Water\ (H_2 0)}$ [5]

This phenomenon has been confirmed by the alkaline treatment have two effects on the fiber:

1. It increases surface roughness resulting in better mechanical interlocking.
2. It increases the amount of cellulose exposed on the fiber surface, thus increasing the number of possible reaction sites [5]. The type of alkali treatment such as KOH, LiOH, NaOH and its concentration will influence the degree of swelling and degree of lattice transformation into cellulose. Alkali solution not only affects the cellulose components such as hemicelluloses, lignin and pectin [6]. Rokbi et al. (2011) [7] Focused on the study of the effect of chemical treatments of fibers by alkalization on the flexural properties of polyester matrix composite reinforced with natural fibers. The used reinforcement Alfa fiber is subjected to NaOH at (1, 5 and 10% for a period of 0, 24, and 48 to 28 ^{o}C). Abdullah (2010) [8] studied the chemical treatment with (Maleic, NaOH, Hcl) solutions at a constant concentration with different times (3, 18, 72, 168, 240 Hours) is conducted in natural fibers (Date palm) are used as reinforcing phase in polyester matrix to form composites. Mechanical properties of these composites such as flexural strength, impact are evaluated Maleic treatment should a relatively good improvement.

This work was carried out to investigate the effect of heat and chemical treatment of sawdust on the mechanical properties and thermal conductivity of unsaturated polyester/sawdust composite material.

Materials and Methods

Materials

The raw materials needed for preparing the composites are:

a. Sawdust

Sawdust is obtained from wood carpentry .The average granular size of sawdust 350- 425 µm.

Table (1): The properties of sawdust which we used in this research

Sawdust name	Type	Origin	Density(g/cm^3)
Jam white	LE	Russia	0.21

b. Unsaturated polyester resin

We have used unsaturated polyester resin (produced by Saudi industrial resin limited, Jeddah, Saudi Arabia) in the form of a transparent viscous liquid at room temperature. The resin is of a thermosetting polymer type that can be turned from liquid to a solid state by adding the hardener. The hardener used is a "Methyl Ethyl Ketone Peroxide" (MEKP).

c. Sodium Hydroxide

High purity sodium hydroxide with assay>99%

d. Cast Mold

The cast mold used for casting the composite specimens, with dimension 30 cm*21cm, consists of two tensile mold specimens and four molds of hot disk specimens. This mold is made of iron and consists of two plates. These plates have been made so that must be free from defects. Before casting the mold was lubricated by Vaseline to facilitate the extraction of specimens.

Preparation of Composites

1. Sieving process to granular size <425µm.
2. Sawdust dried at 80 ˚C for three hours.
3. Determined the weight of materials by using a sensitive balance.
4. The alkali treatment by NaOH at different concentrations 0.5, 1, 1.5 and 2 % (w/v) were done. 10 g of sawdust were immersed in sodium hydroxide solution at different concentrations 0.5, 1, 1.5 and 2 % (w/v) at room temperature, with mechanically stirred for three hours. The mixture was filtered and thoroughly washed with distilled water 8-10 times until obtaining a PH =7 before drying to remove any absorbed alkali. Treated sawdust were then first dried under the sun for a period of 72 hours, then dried by oven at (80°C) for 3 hours to remove moisture.
5. Heat treatment at different temperature (40, 60, 80 and 100 ˚C) and chemical treatment with NaOH at concentrations 2 %(W/V) were done by close system, and mixed by magnetic stirrer for 3 hours. Fig. (1) is a photographic image shows the reflux apparatus used to condensate any vapors resulted from heating process.

Figure (1): Reflux apparatus (Close system).

The sawdust before and after chemically/heat treated at different volume fractions (20, 30 and 40 %) were mixed with unsaturated polyester resin manually until the mixture got homogeneous. The mixture was poured into the mold properly to get the final uniform specimens.

Tensile test

The tension test is a test generally performed on flat specimens. The most commonly used specimen geometries are the dog-bone specimen and the straight sided piecemeal with tabs.

The standard test method ASTM 638-01 is used in this work. The length of the test specimen used is (150 mm). The tensile test was performed in universal testing machine (UTM) with a loading capacity 50 kg. The tests were performed with a cross head speed of (5mm/min). Toughness was calculated by the area under the curve of the scheme (stress-strain) by using Mat. Lab. Math. Works program (Trapezoidal method).

Physical test

a. Thermal conductivity test of sawdust/UPE composites.

The thermal conductivity (TC) demonstrates the thermal properties of developed composites. The test was performed on Hot Disk Thermal constants analyzer device (type: TPS 500, made in Sweden). The samples used in this test were of diameter (40 mm) and (10 mm) thickness.

Mechanical testing

Through the schemes that have been obtained from tensile test which represented of the scheme (stress-strain) and (Load-Extension) curve. Fig. 2 - 33 shows tensile properties were obtained from it.

a. The effect of volume fraction on mechanical properties

The mechanical properties: such as (tensile strength, maximum load), see Fig. (2-9), for raw and treated sawdust decreased gradually with the increasing volume fraction, where the highest value was obtained when V_f=20%, this happen because as the sawdust content increases that's caused an increase in weak interfacial area between the sawdust and the matrix by hydroxyl group in the sawdust. In other hand, the increase of filler content also produced more filler end. This means that there is considerable stress concentrations points taking place by agglomeration of the filler particles and de-wetting of the polymer at interphase aggravates the situating by creating stress concentration points, the poor interfacial bonding causes partially separated micro spaces between sawdust and polymer matrix [9,10], the agglomeration has decreased by well mixing, where the reduction of the volume fraction of sawdust which indicates the ability of sawdust filler to impart greater stiffness to the matrix this is due to cellulose has a high degree of polymerization and crystallinity and is responsible for strength in wood filler and this is the same reason that explains that the increase in the volume fraction of sawdust filler in polymer matrix will require less strain in order to fracture occur Fig.(10-13). For elongation, see Fig. (14-17), the stiffening of polymer chains due to incorporation of sawdust filler reduces the deformability which might be the reason for decrease in % elongation value with increases in sawdust content. The toughness, see Fig. (15-21), was obtained by calculating the area under the curve of the scheme (stress-strain) curve. We can note from that the toughness values decrease with increasing volume fraction of sawdust. This can be explained the higher sawdust content increases the probability of filler agglomeration which results in regions of stress concentrations that require less energy to elongate the crack propagation. In other word, increase in toughness values mean increase in impact strength of composite which is governed by two factors, first, the capability of the filler to absorb energy that can stop crack propagation and second, poor interfacial bonding which induces micro-spaces between the sawdust and the matrix, resulting in easy crack propagation [11, 12, and 13]. Poor interfacial bonding can be enhanced by chemical and heat treatments of sawdust.

b. **The effect of alkali treatment of sawdust on the mechanical properties**

Figs. (9-33) shows the mechanical properties of sawdust/UPE composites at different concentration. Results showed the treatment with 0.5% NaOH does not cause a considerable effect on the sawdust or to the properties of the composites compared to untreated ones, while 1% NaOH improved the mechanical properties of composites, due to better interfacial adhesion between the sawdust and the matrix occurs, imparting improvement in the mechanical properties of the composites. This indicates that interfacial bonding between the filler and the matrix has significantly improved upon alkali treatment leading to increase stress transfer efficiency from the matrix to filler [13] ,but at 2%NaOH we realized the properties began to fall ,this is because at higher alkali concentration , excess delignification of natural filler occurs resulting in a filler weaken or damaged . for toughness ,We can observed that the highest value of toughness when compared between raw and treated sawdust at different concentration ,the highest value of toughness at 1% NaOH and decrease at 2% NaOH, thus the higher impact strengths of treated sawdust / UPE composites, suggest the filler is capable of absorbing energy at 1% NaOH ,because of strong interfacial bonding formed between the sawdust and the matrix as a result of alkali treatment, so this provide an effective resistance to crack propagation. Alkali treatment by NaOH removes certain compounds (e.g. lignin) from sawdust. In result, stress transfer takes place easily and better mechanical properties are attained.

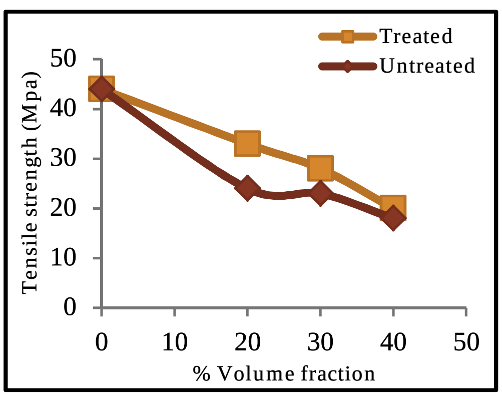


Figure (2): Tensile strength at 0.5% NaOH.

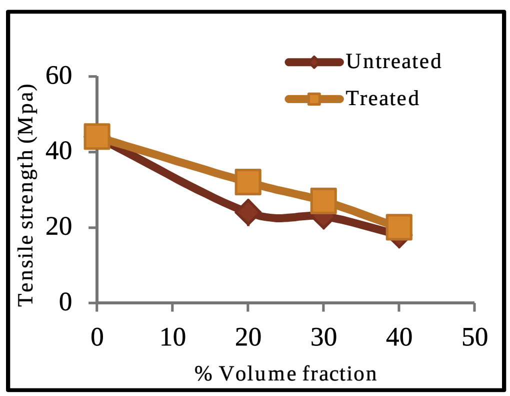


Figure (3):Tensile strength at 1%NaOH.

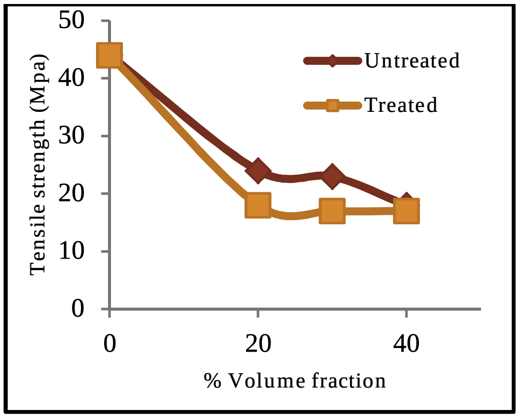


Figure. (4): Tensile strength at 1.5%NaOH.

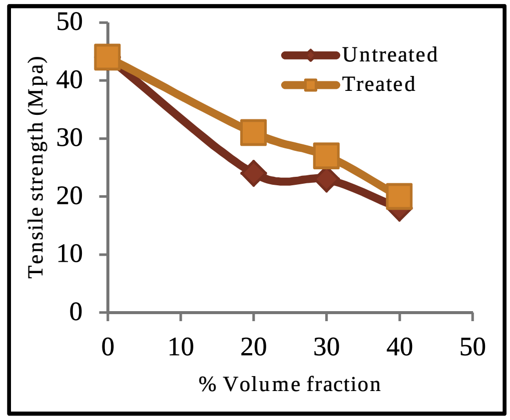


Figure. (5): Tensile strength at 2%NaOH.

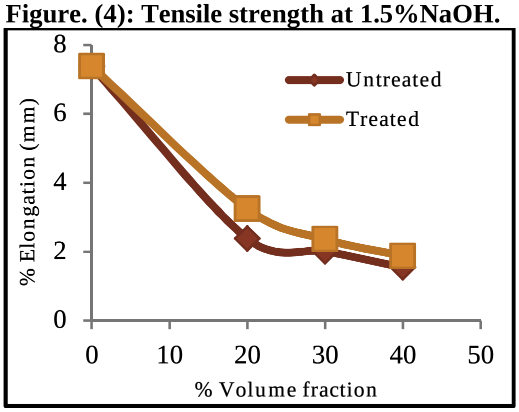


Figure. (6): Elongation at break at 0.5%NaOH.

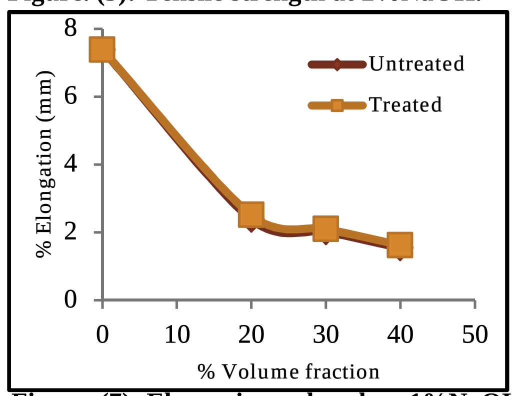


Figure. (7): Elongation at break at 1%NaOH.

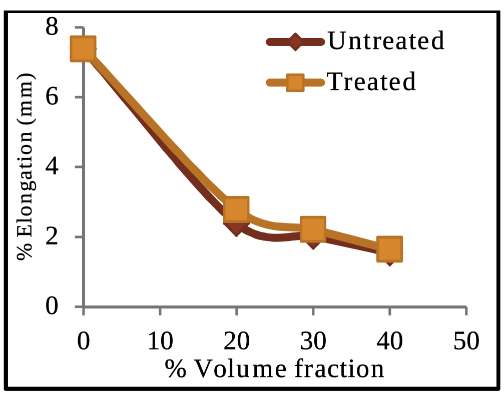


Figure. (8): Elongation at break at 1.5%NaOH.

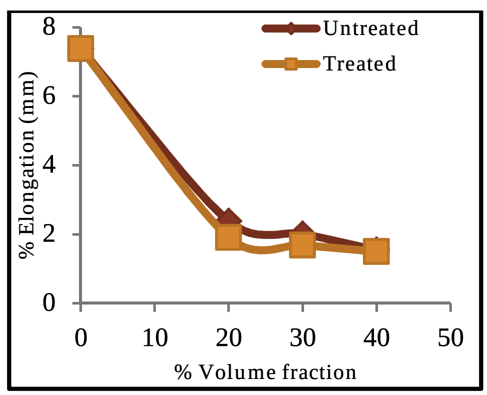


Figure. (9): Elongation at break at 2%NaOH.

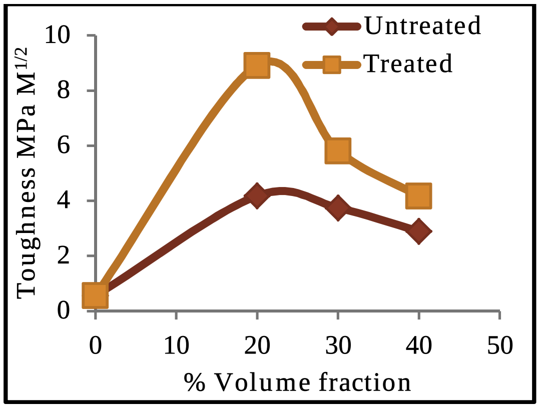
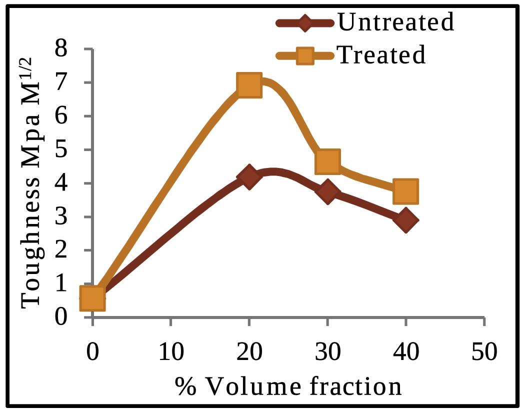


Figure (10): Toughness at 0.5%NaOH.



Figure(11): Toughness at 1%NaOH.

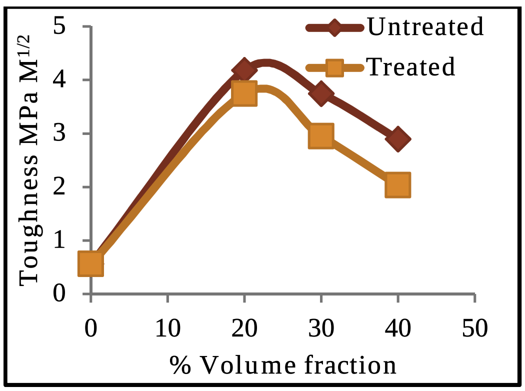


Figure (12): Toughness at 1.5 %NaOH.

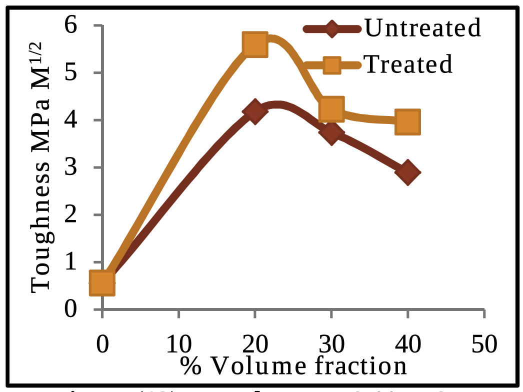


Figure (13): Toughness at 2 %NaOH.

c. **Effect of heat/alkali treatment of sawdust on mechanical properties**

Figs. (14 - 25) shows the mechanical properties results at 2%NaOH.

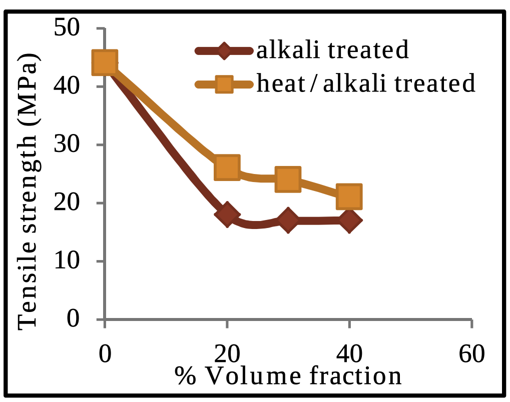


Figure (14): Tensile strength of 2% NaOH/ 40 °c

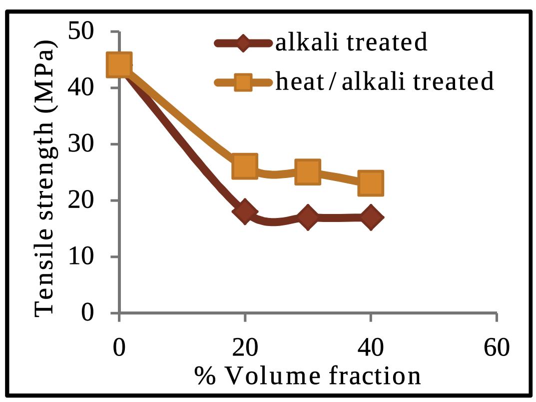


Figure (15): Tensile strength of 2% NaOH 60 °c

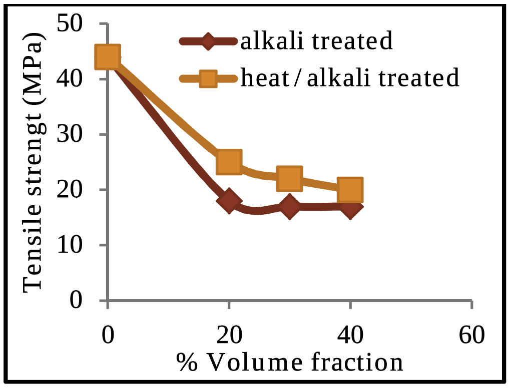


Fig.(16):Tensile strength of 2% NaOH/80˚c.

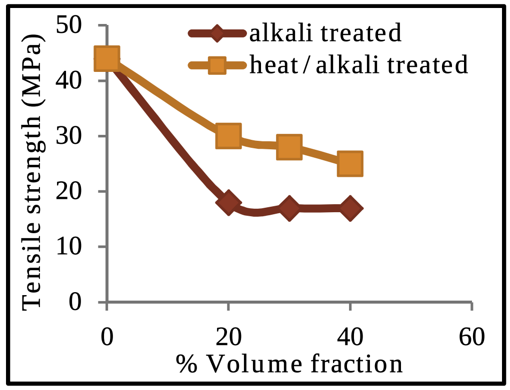


Fig. (17):Tensile strength of 2% NaOH100˚c.

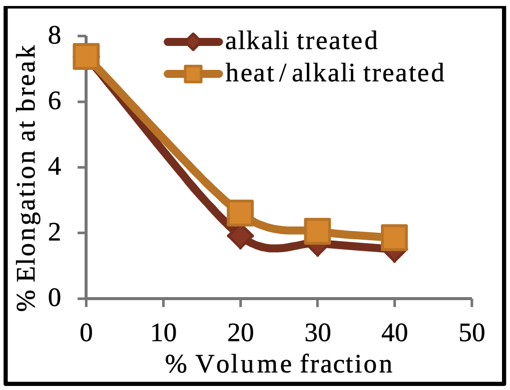


Fig. (18):% Elong. Break(2% NaOH /40 ˚c)

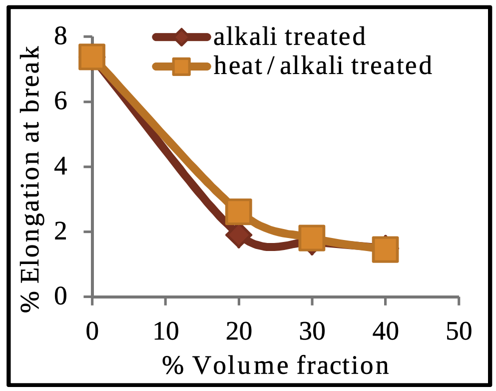


Fig. (19):% Elong. break (2% NaOH/60˚C).

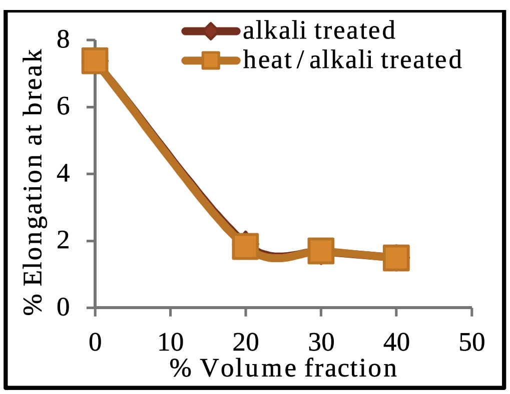


Fig.(20):% Elong. Break (2%NaOH/80˚C).

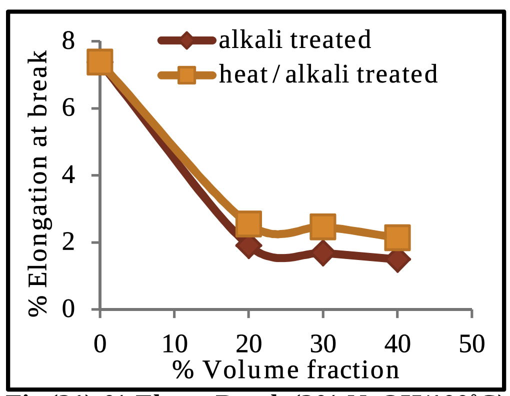


Fig.(21):% Elong. Break (2% NaOH/100˚C)

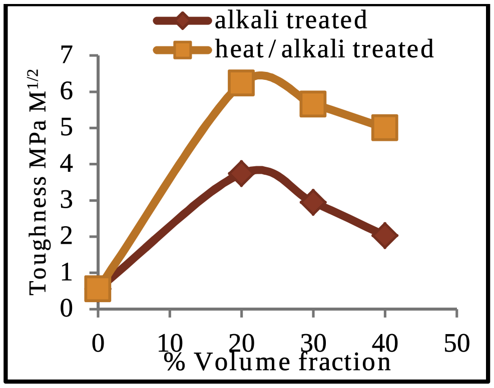


Fig. (22): Toughness of 2% NaOH /40 ˚C

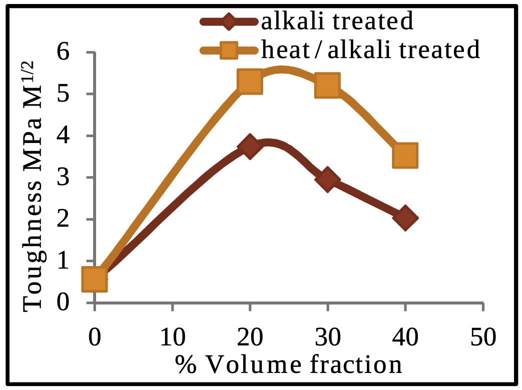


Fig. (23): Toughness (2% NaOH/60 ˚C).

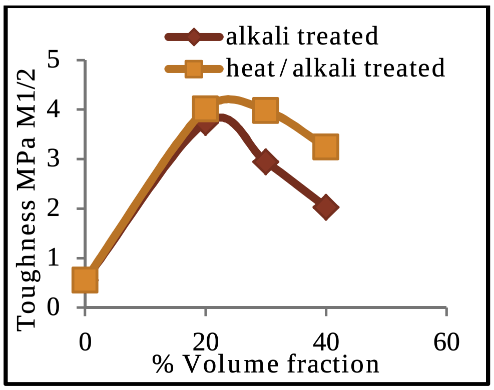


Fig. (24): Toughness (2% NaOH /80 ˚C).

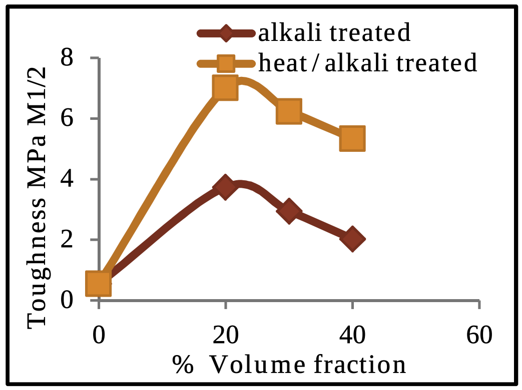
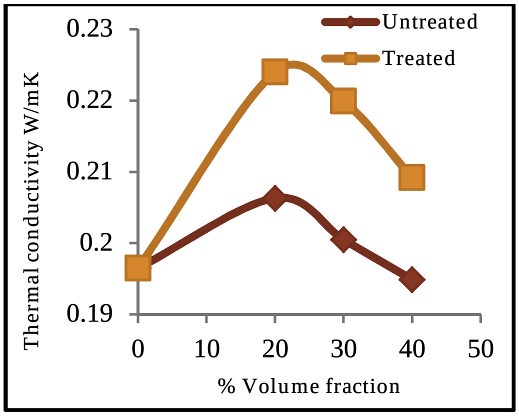


Fig. (25): Toughness (2% NaOH/100 ˚C).

The results showed that the mechanical properties of sawdust were treated with different temperature 40 ,60 ,80 and 100 ˚C at 2% NaOH increased at increase heat treatment at 40,60 and 80 ˚C, while decreased at 100 ˚C. The maximum value was found at 80 ˚C which could attributed to the increase of crystallinity index of sawdust when the heat/alkali treatment was done ,while the minimum value ,this is considered that the thermal degradation of sawdust may be occur at 100 ˚C [15].

Thermal conductivity:

The results of thermal conductivity were obtained from hot disk thermal analyzer constant device, Fig. (26-29) are listed below represent thermal conductivity test results.



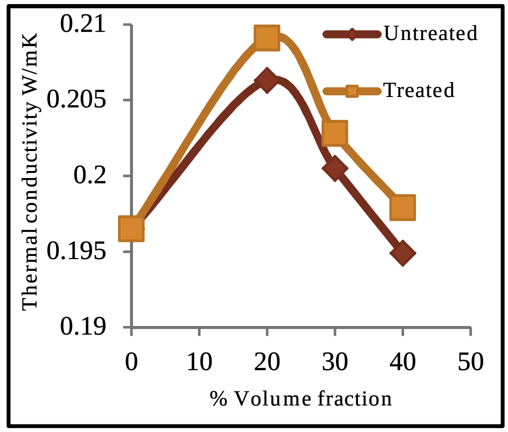
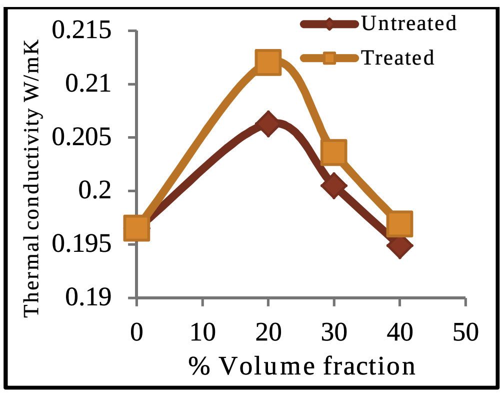


Fig. (26): Thermal conductivity at 0.5% NaOH. **Fig. (27): Thermal conductivity at 1% NaOH.**



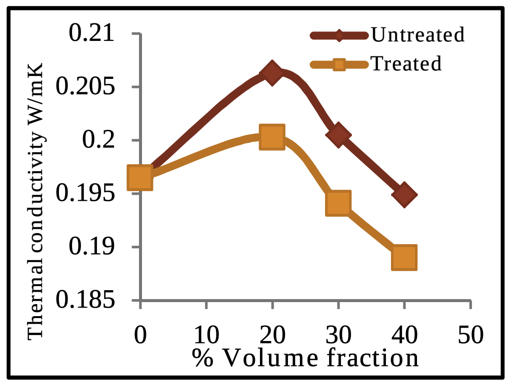


Fig.(28):conductivity at 1.5% NaOH. **Fig. (29): Thermal conductivity at 2% NaOH.**

a. The effect of volume fraction of sawdust on thermal conductivity

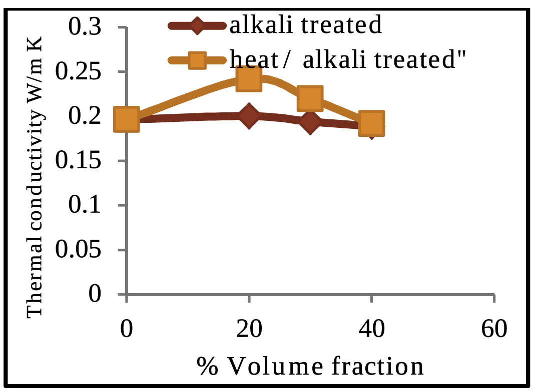
It can be seen that from Fig. (24-33) V_f = 20% of sawdust reinforced polymer matrix exhibit high thermal conductivity followed by 30 and 40 % volume fraction of sawdust this is caused to the following reasons:

1. As more sawdust reinforced into the polyester matrix, the heat conductivity of the composite material decrease which in turn provide the integrated fiber orientation in the matrix. This feature can be taken into consideration in designing the house- hold products / components which are subjected to heat and temperature [16].
2. Increase of the sawdust content caused increase the number of micro-voids in the composite is caused by the bigger amount of poor bonded area between sawdust and polymer matrix , so when thermal energy will move through matrix material by the vibratory motion of the atoms so when the atoms reach to interface region the medium will differ which will move through it , and this causing obstruction to the transition of thermal energy and thus will decrease thermal energy transmitted values and will cause drop of thermal conducting values.

b. The effect of alkali treatment of sawdust on thermal conductivity

From above figures can be noted that 0.5% NaOH (w/v) was not a significant influence on thermal conductivity, on the contrary, thermal conductivity is the maximum for composite prepared from 1% NaOH treated fiber this can be attributed to the surface area of the fiber increases due to the dissolution of lignin, hemi-cellulose and other substances associated with the fiber. This results in large area of the contact between the fiber and the matrix leading to increase in thermal conductivity. It can be observed from Fig. (29), that, the thermal conductivity decreases when the sawdust was treated with 2%NaOH. This is because at high concentration of NaOH led to an increase in amorphous cellulose content at expense of crystalline cellulose, so when the crystallinity decrease the thermal conductivity decrease too [17].

Thermal conductivity of heat/alkali treated sawdust/UPE increased at (40, 60, 80 and 100 ˚C. The maximum value of thermal conductivity found at 80 ˚C due to increase the crystallinity at this temperature in contrast at 100 ˚C the absence of crystallinity caused decreased of thermal conductivity.



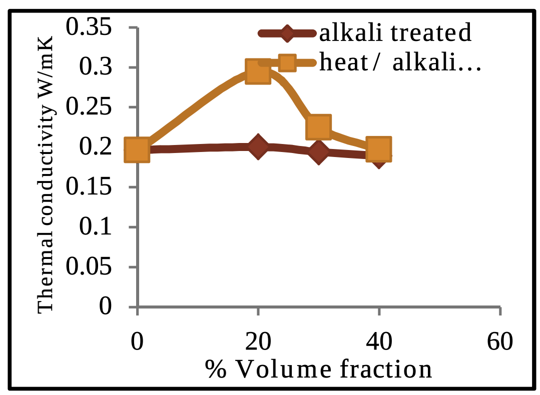
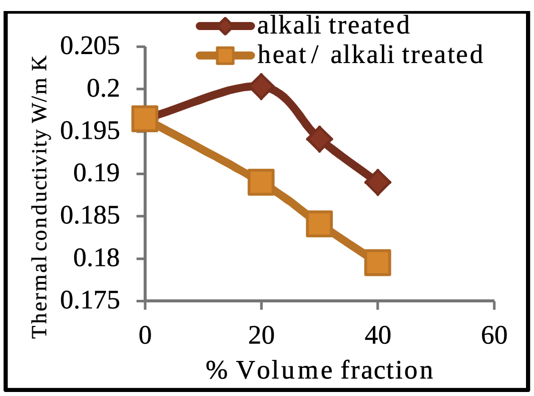


Fig. (30): Thermal cond. (0.5%NaOH/40˚C).
Fig.(31):Thermal cond. (0.5%NaOH/60˚C).



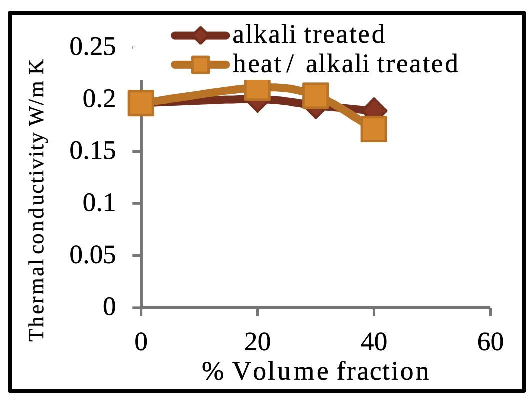


Fig. (32): Thermal cond. (0.5%NaOH/80˚C) **Fig. (33): Thermal cond. (0.5%NaOH/100˚C)**

CONCLUSIONS

For the purpose of improving mechanical and thermal insulating properties of sawdust/UPE composites, alkali treatment with NaOH at 0.5,1, 1.5 and 2% (W/V) were done, so when the mechanical properties decrease at 2% NaOH heat/alkali treated of sawdust were performed at 2% NaOH with different temperature 40, 60, 80 and 100 ˚C and the effect of volume fraction of sawdust on tensile properties and thermal conductivity were investigated. The obtained results are:

1) For alkali treated, the mechanical properties of sawdust/UPE at 1%NaOH for %Vf =20 showed the highest value, and thermal conductivity decreased at 2% NaOH at %Vf=40.

2) For heat/alkali treated, the mechanical properties show the maximum value at 80 ˚C, and thermal conductivity decreased at 100 ˚C

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