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Synthesis of composites based on waste natural Products and Polyurethane

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Abstract

In this study, new polyurethane composites have been prepared by the treatment and functionalized of waste leave sidr powder with polymeric methylene diphenylene diisocyanate (PMDI), and the mixing effect on the properties of these composites were evaluated using different weight percentages of carbon fibers (CFs). Thermal treatment of the prepared natural composites was done at different temperatures, where the studies used the XRD and SEM showed the heat treatment at 900°C, lead to formation of Nanocomposites. On the other hand, the flammability test of these composites and hybrid composites (containing different % of CF) were a analysed using UL-94V methods, the results show, that these prepared composites were flammable and fire resistant. Also, the effect of CF on the ablation properties of these composites was evaluated using an oxygen-acetylene ablation device, and along with the increasing the content of CFs, the mass and linear ablation rates both decreases, while the insulations index were increased.

Keywords: Polyurethane Composites; Carbon Fibers; XRD; SEM; Ablation; Flammability.

1. Introduction

Composite materials are made from two or more constituent materials with significantly different physical or chemical properties (1, 2), that when combined produce a material with characteristics different from the individual components.

The interest in producing natural product-reinforced polymer composites has gained considerable attention in the last few years due to sustainability and degradability issues (3-5). Fiber-reinforced polymer (FRP) is a composite material made form a polymer matrix reinforced with fibers. The fibers are usually glass, carbon, or aramid, although other fibers such as paper or wood or asbestos have been sometimes used.Natural products have been introducing as it's or as fibers with a view to producing light weight composites, along with reduced prices relative to conventional synthetic reinforced materials like Kevlar or fiber glass, as well as to produced green-reinforced polymer composites (6). On the other hand, natural products-reinforced polymer composites have outstanding and comparable thermal properties to synthetic reinforced materials, leading to extend their application for engineering materials such as automotive, aerospace industry and construction structure (7). Hence, the formation of polymer matrix-based composites

(PMCs) has remained one of the most efficient methods to influence the properties of polymers. Composite materials have been used more widely in engineering and product applications in the last decades, and this trend continue. Among the major benefits these materials offer is the high stiffness to weight ratio, which makes a strong case for the transportation industry, and the good durability, which has supported the use of composites in aggressive environments. Polymers and composite materials are often exposed to environmental influences such as water, humidity, elevated temperatures, pH, mechanical stress, and their combinations. Thus, environmental factors negatively impact the performance, affecting their durability (8). In the original definition of composite materials, two basic constituents are considered, namely, the matrix and the reinforcing phase. In terms of the amount of each of them, for the most part, the matrix is in greater proportion as compared to the reinforcement, which in turn acts as the "guest phase" and the matrix, as the host (9-11). By-products from the agricultural industry are generally the source of bio-based fillers, which can be applied to the production of polymer-based composites (12, 13). Several different chemical modifications were proposed for the bio-based fillers, e.g., mercerization (using sodium hydroxide), acetylation (using acetic

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anhydride and acetic acid), oxidation (using potassium permanganate), grafting (by ring opening polymerization, living polymerization or using coupling agents), and others. The procedure of single chemical modification can be different for different types of bio-based fillers and it results from the different chemical composition, size, surface characteristic, and mechanical properties of filler subjected to the process. Several different bio-based fillers were proposed, for example: hemp fiber, flax fibers, kenaf fiber], sugar palm fibers, roselle fibers, tea waste fibers, and cocoa pod husk fibers. Rice and buckwheat husks are examples of agriculture by-products that can be applied as a filler in the preparation of polymer composites (14, 15). On the other hand, the ablative properties of most polymers ablative composites (i.e., resistance to thermochemical corrosion and mechanical scouring as well as thermal stress caused by high-temperature and high-speed heat flux) are mainly determined by the structure of the polymer layer (16, 17). Carbon fibers (CF) are marvelous materials that can be employed in various important engineering applications. Carbon fibers possess outstanding physical and chemical properties; for instance, their high tensile strength combined with a low density makes CF ideal candidates to be employed in high strength low weight structural parts (18). The main objective of this research is the synthesis of polyurethane composites using waste leave sidr and effect of carbon fibers (CFs) weight percent on the flammability and ablation properties on the prepared composites.

2. Materials and methods
2.1. Materials

Polymeric methylene diphenyl diisocyanate (PMDI), was supplied from Sigma Company with the following specification:
-Appearance: Dark Brown liquid
-NCO content (wt %): 30.0-32.0
-Viscosity (cps/250c): 115-120
-SP. Gravity (250C): 1.23-1.25
-Acid (wt. %): Max 0.1
Carbon fiber: chopped carbon fiber as a short, thin, Flak type was supplied from with the following specification:
-Density (g/cm3): 1.80
-Fiber length chopped (mm/in): 6.0/0.24
-Tensile strength (Gap): 4.0
-Elongation at break (%): 1.7
-Sizing type: Epoxy resin.
Ziziphins leaves were collected from the buck thorn tree, washed well and exposed to the sun in order to dried then crushed to powder 200-300, micron in size. (14)

2.2. Instrumental

-FTIR: measurement of the product was conducted on IR Shimadzu-8400 spectrophotometer (Japan), Solid samples were examined using KBr disc. The samples were carried out at Basrah University /college of science-chem.dep.

-SEM: scanning electron microscopy was used to study the morphology of the samples. Beam voltage of 20Kv. prior to the SEM investigation, the samples were sputtered with gold at room temperature.

-XRD: X-ray diffraction analysis were done with Brokers XRD, D8-device with a nickel-copper filter (copper Kα λ=1.5406 A0), and the measurement of prepared Samples was carried out at Basra university /college of science-phys.dep.

-UL-94 V: Flammability test for pure poly urethane and treated polymer with different percentage of carbon fiber was studied by a vertical uL-94 test according to ASTM D3801. The dimension of the specimen was 125mm Long, 13mm wide, and 3 mm thick.

The samples were done vertically over surgical cotton and ignited with Bunsen burner the test types were classified on the rating presented in Table (1). V-2 and the lower and of samples was exposed to the flame for 10 seconds and then removed. The times were recorded.

Figure 1 a) Carbon Fibers, b) Ziziphins leaves powder

Table 1: Burning criteria for UL-94 vertical rating

Test criteria	UL-94 V rating		
	v-0	v-1	v-2
Burning time of samples (second)	≤ 10	≤ 30	≤ 30
Dripping of burning samples (ignition of coating)	No	No	No
Specimen completely burned	No	No	No

-Ablative test: It was done by oxyacetylene torch according to ASTM E285-80. The dimension of the samples was accurately measured and the conditions used in this test are listed in table (2).

2.3. Method

Samples of polyurethane composites were prepared by mixing of MDI with leaves powder in the ratio 2PMDI: 1 Sidr leaves powder (wt. /wt.) at room temperature for 5 min, then cure cycle was done to get final products, 48 h. at room temperature, 24 hours at 60^0C, 24 hours at 100^0C and then for post cure 4 hours at 120^0C. In the case of carbon fiber, different weight percentage of carbon fiber were used as donated in the table 3. And the Same procedure as above were done to prepare the polyurethane composites contain carbon fiber.

Table 2. Representative specification required for ablative tests.

| Conditions	Required
Dimension of the specimens	100 X 100 mm in size and 7 mm thickness.
Flow rates of oxygen and acetylene	1.36, 1.04 m^3 \ h. respectively
Burner torch specification	2mm diameter
Torch flame temp	Around 2400 ^{0}C
Distance between nozzle and the specimen	20 mm
Contact angle between the torch flame and specimen surface	90 ^{0}

Table 3: Percentage of compounds prepared for polyurethane composites.

| Sample No.	Leaves powder %	Carbon fiber %	Weight ratio of MDI: Another component was
A1	100	0	2:1
A2	75	25	2:1
A3	50	50	2:1
A4	25	75	2:1
A5	0	100	2:1

3. Result and dissection

3.1. FTIR analysis

Through the spectrum of Leave sidr powder as shown in Figure 2. Broad and strong absorption band was observed at 3383 cm-1 due to the hydroxyl groups present in their structure of leave sidr (19), also a positive test of the phenolic hydroxyl groups present in leaves sidr was observed by using ferric chloride reagent. So new polyurethane was prepared thought direct condensation polymerization between hydroxyl group present in their structure of leave sidr powder and the isocyanate group in PMDI, scheme 1 show the reaction route.
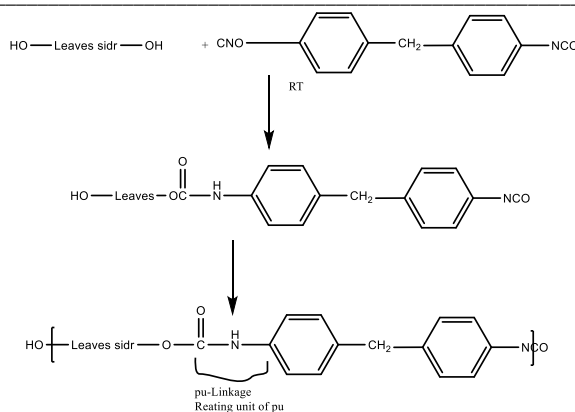
Scheme 1. Polyurethane composite synthesis.

The FTIR of the prepared polyurethane are shown in Figure (3, 4). Absorption band at 3392 cm-1 related to the stretching vibration of N-H bond present in the urethane groups, and included of some unreacted hydroxyl groups. Also, absorption bands at 2920 and 2852cm-1 due to symmetric and asymmetric stretching vibration of C-H aliphatic bond of CH2 groups. On the other hand, absorption band at 2275 cm-1 due to unreacted isocyanate (NCO) groups (20,21) were only observed in the spectrum in Figure 2, and this band nearly disappear after complete a curing as shown in Figure 3. Bands at 1653-1716 cm-1 indicates the presence of carbonyl groups in urethane linkage. The stretching vibration of C-N bond was observed at 1230 cm-1, and the multiple bands in the range (1014-1101) cm-1 was due to C-O bonds in flexible segments.
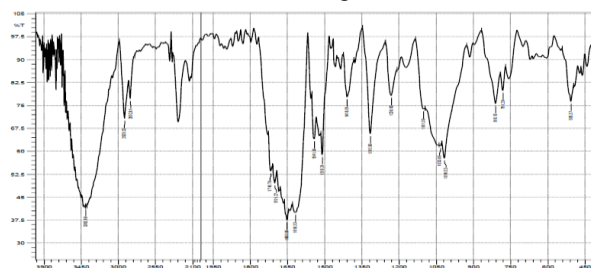
Figure 2. FTIR spectrum of waste leave sidr powder.
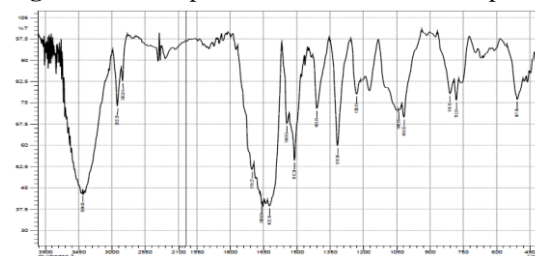
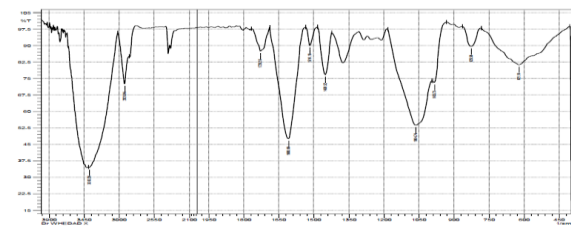**Figure 3.** FTIR spectrum of the prepared polyurethane composites before complete curing.

Figure 4. : FTIR spectrum of the prepared polyurethane composites after post curing.

3.2. X-RD study

To evaluate the influence of heat treatment of the prepared polyurethane donated (A1) at different temperature (500,700 and 900^{0}C) on the crystalline structure of the polymers. X-ray diffraction brokers is performed to study the structural of the polymers. The diffraction angular 2ɵ is ranged from 50 to 800 with an increment of 0.01. The diffractometer system uses cu tube as an X-ray source with an intensity of 40mA and a tension of 45 Kv. The XRD patterns of the samples were shown in figures (5) it is observed through the overlap of the peaks, the absence of any crystallization in the polymer before heating i.e. abroad peak around (2ɵ =20) with d-spacing 4.4 A0. While for the polymer after heat treatment at 500^{0}C, a change in the XRD spectrum was observed (Fig.6), the peaks began to reduce overlaps and aggregate, but the polymer still amorphous. Only the presence of aspirating peak at position (2ɵ =49) and at position 2 θ (25-30). For the polymer after heat treatment at 700^{0}C, the XRD spectrum shown in Figure (7), indicate that the polymer was crystallize during the heat treatment and new single peaks in different locations was appear, the highly intensity peak at (2ɵ= 27). The best crystallization rate of the polymer was obtained after heat treatment at 900^{0}C, as shown in Figure (8), where the peaks appear more separated and clearer, in addition to the intensity of more than 100. Also new peaks were emergence which indicates the crystallization of some other aggregates that were not crystallization at a temperature 700^{0}C, as show anew peak at (2 ɵ=29). Tables (4-7), show the peak list of the polymer obtained from XRD analysis.
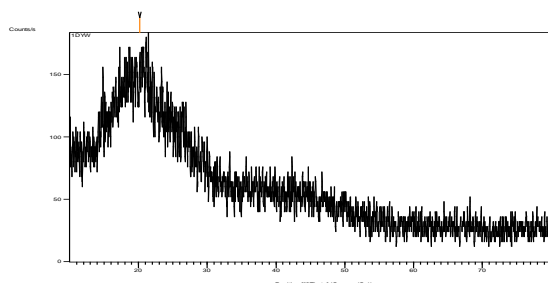

Figure 5. XRD spectrum of the prepared composite before heat treatment.
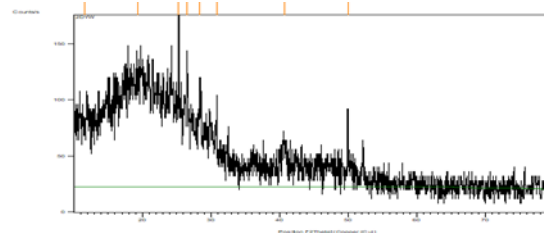

Figure 6. XRD spectrum of the prepared composite after heat treatment at 500 C^0.

Table 4. Characteristic value of XRD results.

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
20.1641	22.82	2.3040	4.40025	100.00

Table 5. Characteristic value of XRD results.

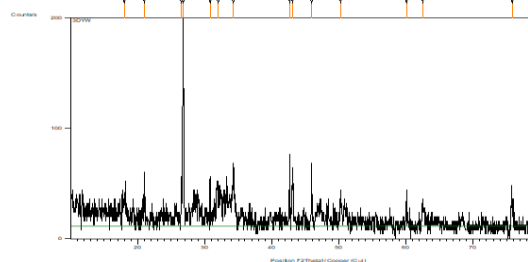| Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
11.5722	15.04	1.2595	7.64706	42.94
19.2748	23.89	3.7786	4.60503	68.23
25.3037	35.02	0.1181	3.51983	100.00
26.5147	28.27	0.2362	3.36176	80.72
28.4089	22.00	0.3149	3.14178	62.84
30.8275	20.49	0.1378	2.90058	58.52
40.7062	8.94	0.6298	2.21658	25.53
49.9465	11.58	0.2880	1.82451	33.08

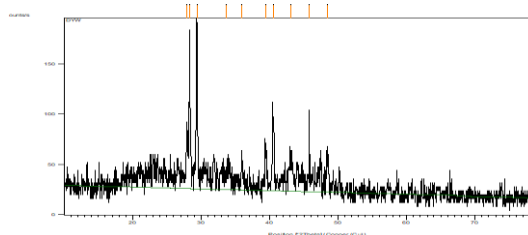Figure 7. XRD spectrum of the prepared composite after heat treatment at 700 C0.

Figure 8. XRD spectrum of the prepared composite after heat treatment at 900 0C.

3.3. EM-study

Scanning electron microscopy can be used to characterize the polymers and to examine changes in the microstructures and morphology during synthesis of the polymers. (22, 23).

The SEM micrographs of heat treatment polyurethane were done and are shown in Figures (9-12) The polymer before heat treatment (Fig.9), show large agglomeration as shown in size 100KX and 200kx, these formed aggregates indicate that the polymer is amorphous, as the percentage of deformed

is very clear, and the morphology of the polymer is non-Nano material. For sample polymer heated at 500^0C for 1hrs. Figure (10), shows the morphology of the polymer and the same behavior for the polymer before heating, also the agglomeration also appears indicating that the polymer did not crystalline at this temperature.

On the other hand, Figure (11) show the micrographs of the polymer heated at 700^0C for one hr. the particle appears to be separated and the clumps disappear, meaning that the polymer begin to crystalize in a polyhedral as shown in size 1100Kx and 200Kx with the diameter ranging from 37.09nm to 45.01nm. Finally, the polymer heated at 900^0C. Figure (12) show the transformation of the polymer into a nanostructure material at this temperature, and as the crystallization rate is very high and the clumps disappear completely. And the particles here are better separated, homogeneous, and evenly distributed. The shape of the particles is polyhedral as shown in sizes 100Kx and 200Kx with a diameter ranging from 191.86 to 273.47 nm.

Table 6. Characteristic value of XRD results of composite after heat treatment at 700 C^0.

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
18.0275	6.19	0.4723	4.92072	13.13
20.9898	7.55	0.2362	4.23247	16.00
26.5392	11.39	0.1771	3.35872	24.15
26.7862	47.15	0.1181	3.32830	100.00
30.7679	11.25	0.1181	2.90606	23.86
32.0599	6.88	0.6298	2.79183	14.60
34.2625	11.13	0.2362	2.61723	23.61
42.6520	13.22	0.1181	2.11986	28.04
43.0875	11.29	0.2755	2.09943	23.96
45.9576	9.44	0.1181	1.97478	20.03
50.2910	6.84	0.2362	1.81432	14.51
60.0841	7.12	0.2362	1.53992	15.11
62.5377	4.89	0.3936	1.48527	10.37
75.8365	4.65	0.3840	1.25346	9.85

Table 7. The characteristic value of XRD results of the composite after heat treatment at 900 0C.

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
27.9466	15.32	0.1378	3.19269	37.66
28.3237	39.04	0.0984	3.15103	96.00
29.3885	40.67	0.1378	3.03925	100.00
33.6928	4.46	0.9446	2.66017	10.98
35.9822	7.47	0.2362	2.49600	18.36
39.4253	9.46	0.2362	2.28559	23.26
40.5124	17.12	0.2362	2.22674	42.10
43.1206	8.04	0.4723	2.09790	19.78
45.7919	20.33	0.0590	1.98155	50.00
48.4734	9.32	0.3840	1.87646	22.93
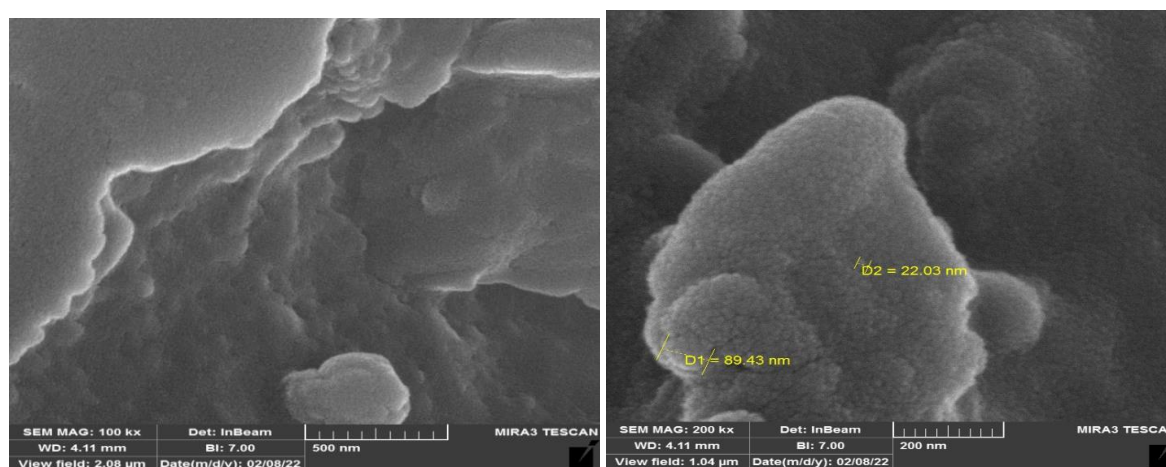
Figure 9. SEM image of the prepared composite before heat treatment.
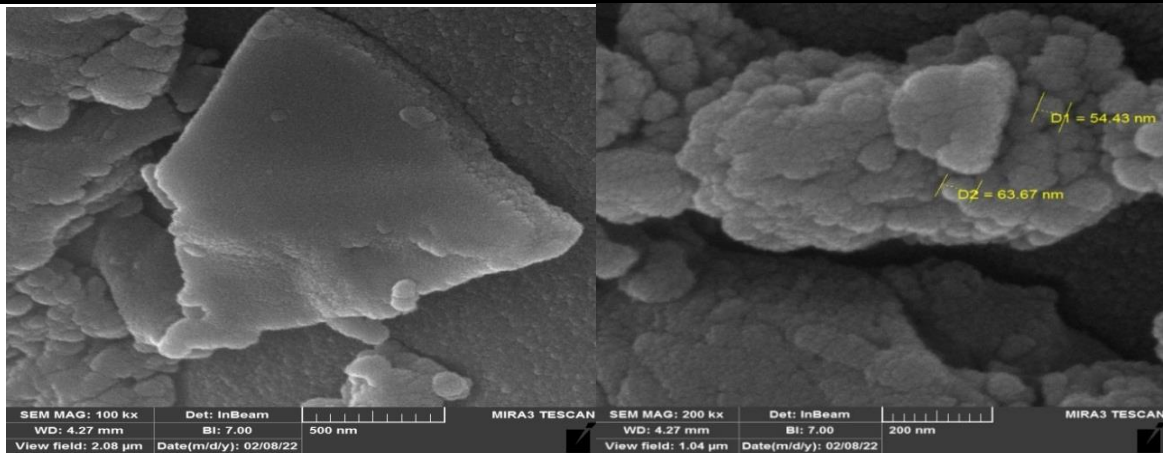
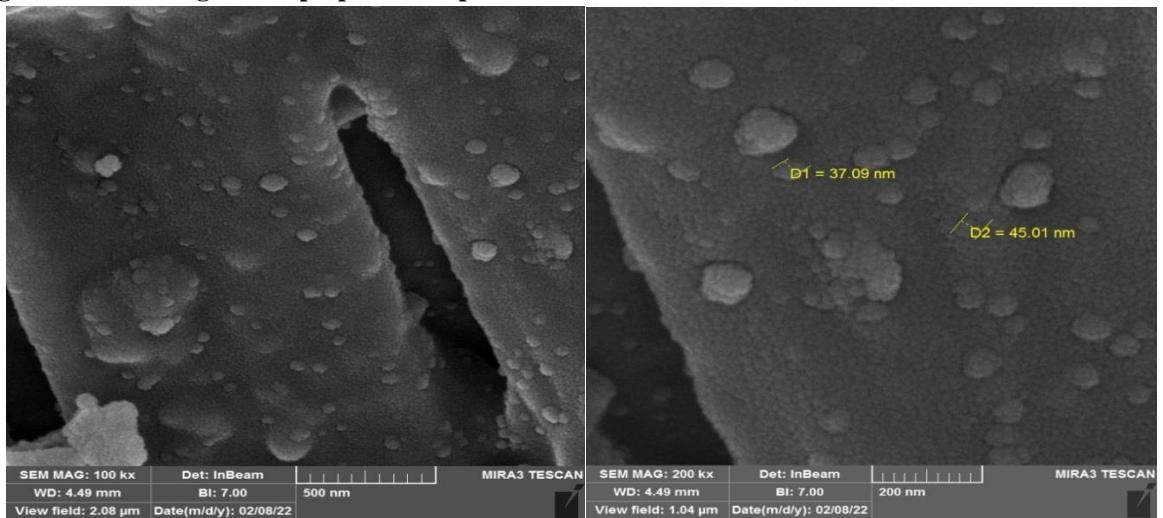**Figure10. SEM image of the prepared composite after heat treatment at 500 0C.**

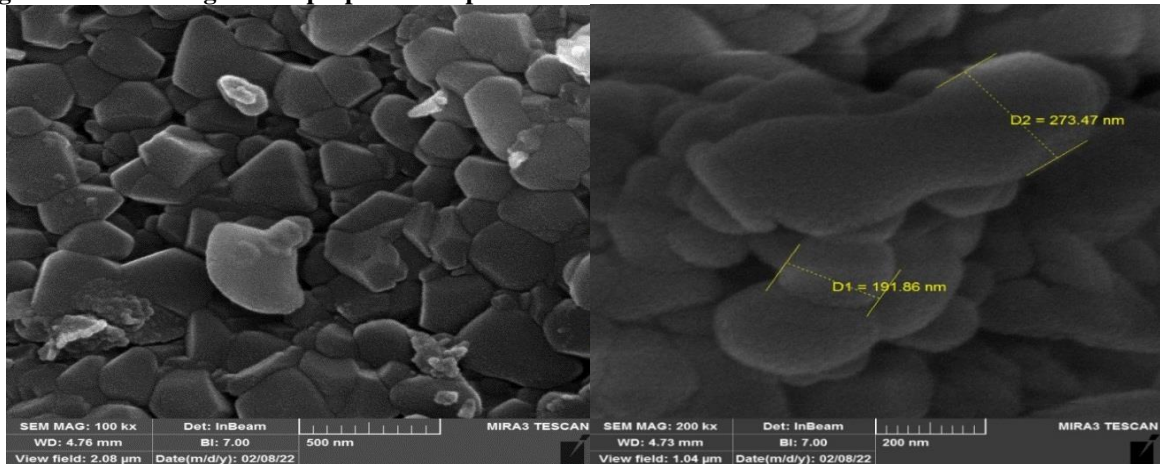**Figure 11. SEM image of the prepared composite after heat treatment at 700 0C.**

Figure 12. SEM image of the prepared composite after heat treatment at 900 0C.

3.4. The thermal Insulating characteristics

-Material: The burning test and ablative characteristics were evaluated for the following prepared polyurethane composites (A1-A5) after complete cure.

-Specimens: The test specimens were prepared by casting the required polymer in wood molds (dimension 16 cm length, 5cm thickness and 7cm width, Figure 13 a, b). The samples were cured under pressure as above in experimental. Then cut the sample according to their test for burning (24, 25) with dimension 127 X 13 X 10 mm3 (ASTM D 3801-96), while for ablative test the specimens were prepared by casting the polymers in paper molds (dimension).

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3.5. vertical burning test UL-94

This test measured the self-extinguishing time of the vertically polymer specimen. The top of the test specimen is clamped to a stand and the burner is placed directly below specimen (Figure 14). The flame is brought into contact with test specimen for 10 seconds, after which the burner is remover. The flame should be a blue flame and 20 mm in high, v-rating can provide the so-called v-0 rating. The material was rated v-0 if the flame extinguishes within 10 seconds after removal of the burner. The v-1 and v-2 rating required that the flame extinguish after 30 seconds after removal the burner. So the results obtained from this test was listed in table (8) .and the results shown that all the prepared samples with different ratio of carbon fibers give the same behaviors. i.e all the samples were self-extinguishers.

Table 8. Test result of the prepared composites.

Sample	Flaming drops	Cotton ignited	UL-94V Rating
A1	No	No	V0
A2	No	No	V0
A3	No	No	V0
A4	No	No	V0
A5	No	No	V0

From the result, the composite polymer without carbon fiber the flame distingue with 2 second, with different percentage of carbon fiber the flam distingue after 5-7 second for all samples. In general, all the prepared composites in this study were flame retardant.
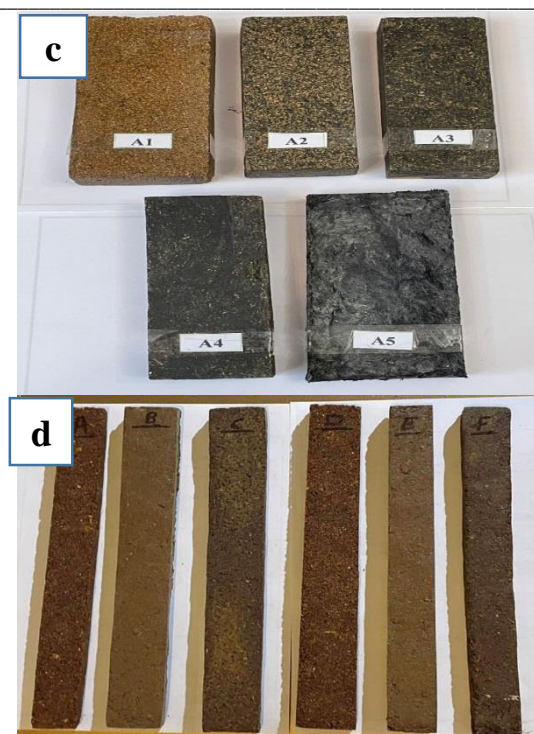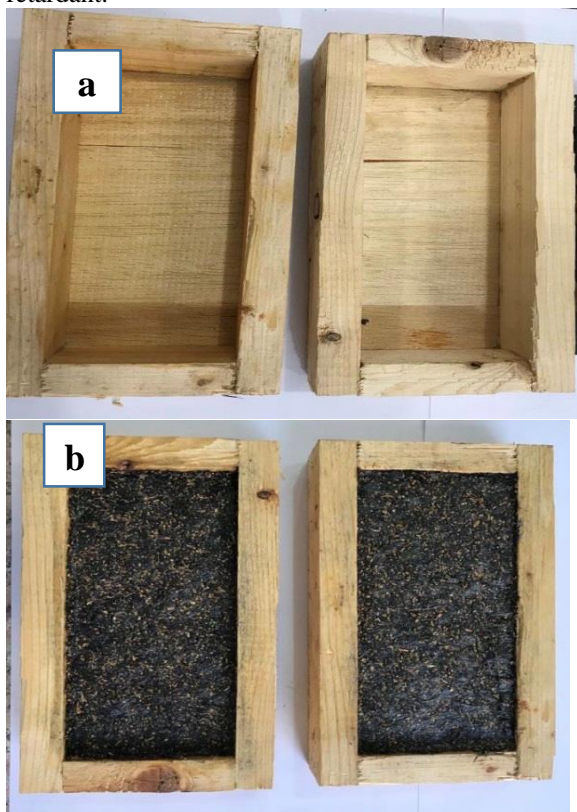
Figure 13. a) Wood molding, b) molding of the composites, c) composites material after complete curing and d) samples for UL-94V test.
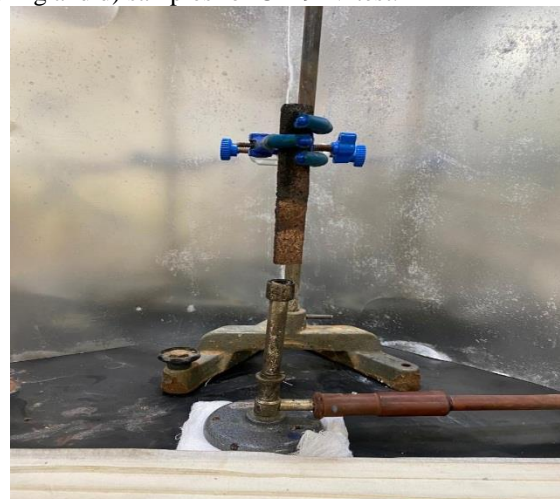
Figure 14. Set up apparatus for UL-94V test.

3.6. Ablative tests:

The ablative tests were carried out according to ASTM No. E285-80. (26, 27). A schematic diagram for the set-up used is shown in Figure (15). All the measurement was carried out at chem-Dep. /college of science-university of Basra. The dimension of the specimens was accurately measured and the conditions used in this test are listed in table (9). Then the samples (A1-A5) were subjected to oxy-acetylene torch and the temperature at the back face of the sample was measured instantaneously by an accurate contact digital thermometer 0.01 0. Form this study linear and mass ablation rates insulation

index and, char yield was calculated according to the Eqs.1-4.

1. Linear ablation rate (LAR) (mm/s) = T_0-T_I/t
2. Mass ablation rate (MAR)(g/s)   = M_0-M_I/t
3. Insulation index (s/m)   = $I_T = T_t/T_0$
4. %char yield   = $(M_0$-$M_I/M_0)$
   *100

Where T_0, M_0, T_I and M_I are the thickness and mass of the ablator samples before and after ablation testing, respectively, it is the ablation time fixed at 30s, and tT is the burn through time (s) representative the time required to completely pierce the schematic illustration for calculating linear ablation rate. The results obtained are summarized in Table (10), and Figures (16-19). From the results, the increase of carbon fibers in the polyurethane composites leads to decrease in the LAR from 0.401 mm\s to 0.068 mm\s when the carbon fiber increase from 0 wt % to 100 wt %., and the same behavior for MAR results. This result is in line with ref.28. In addition increase in carbon fiber content in polyurethane composites leads to increase in insulation index that means the incorporation of carbon fibers in the prepared composites enhanced the ablation performance.

**Table 9. Representative specification required for ablative tests.**

| Thickness of the specimenl: 7mm |
|---|
| Diameter of the specimen: 100 mm |
| Oxygen -Acetylene mole ratio: 17:01 |
| Nozzle diameter of the torch: 2mm |
| Distance and angle between the specimen surface and the torch: 20 mm, 900 |
| Flow rates of oxygen and acetylene: 1.36 mm3\h and 1.04 m3 \h |
| Torch flame temp: 2400^{0}C |
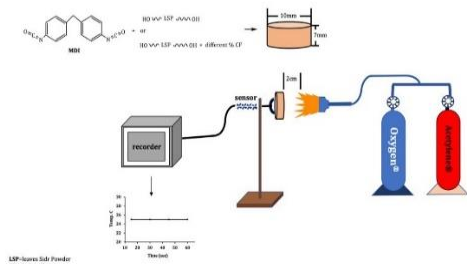
Figure 15. Schematic diagram of oxy-acetylene torch ablation test.

**Table 10. The ablative characteristic of the prepared polyurethane composites.**

| Composite Sample	LAR (mm\s)	MAR (g\s)	Char yield %	Insulation Index (s\m)
A1	0.401	0.080	9.32	3.46
A2	0.183	0.073	8.18	4.28
A3	0.130	0.054	6.96	5.62
A4	0.117	0.045	5.59	5.75
A5	0.068	0.032	5.02	6.03
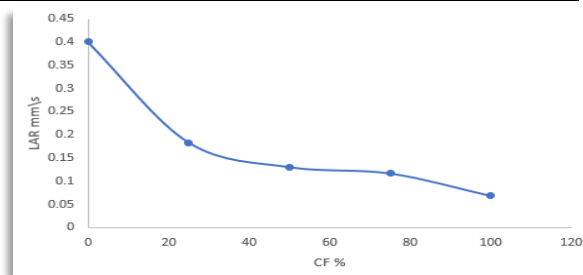
Figure 16. Linear ablation rate of the polyurethane composites at different percentage of carbon fibers.
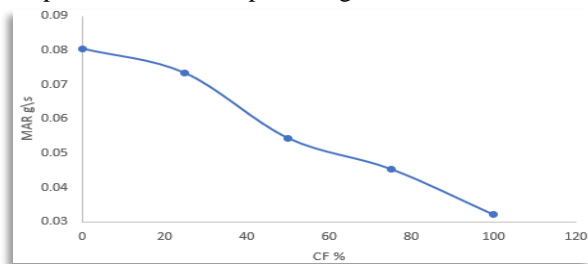
Figure 17. Mas ablation rate of the polyurethane composites at different percentage of carbon fibers.
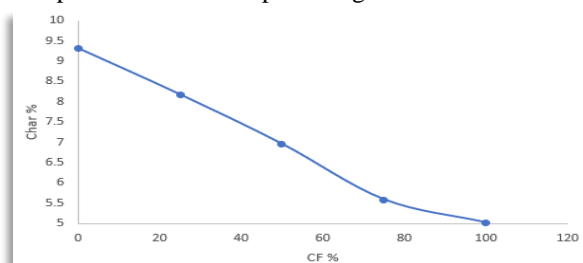
Figure 18. Char yield of the polyurethane composites at different percentage of carbon fibers.
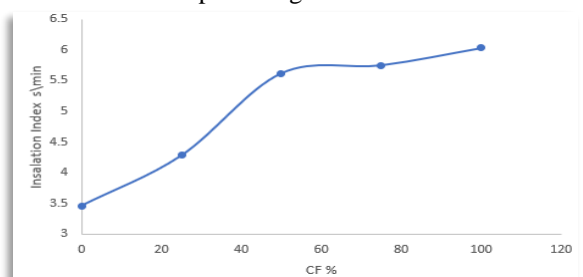
Figure 19. Char yield of the polyurethane composites at different percentage of carbon fibers.

4. Conclusions

Polyurethane composite based on waste leave sidr was synthesized, and hybrid composites also were prepared using different weight percentage of CFs. Nano composites was formed after heat treatments of the composites based on waste leaves sidr at 90^0 C. On the other hands all hybrid prepared composites in vertical UL-90 test showed better properties and the ablation study show the increasing of CFs contents leads to increases insulation index and decreases both LAR and MAR..

Conflicts of interest

There are no conflicts to declare.

Formatting of funding sources

Nill.

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