Epoxy nanocellulose paper

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Preparation of Epoxy-Nanocellulose Composite Material from Iraqi Sugarcane Bagasse Residue

Preparación de material compuesto de epoxi-nanocelulosa a partir del residuo de bagazo de caña de azúcar iraquí

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ABSTRACT/ In this study, composite of epoxy-nanocellulose resin from the agriculture waste (Sugarcane Bagasse residues) available locally in Iraq synthesized. The nano scale polymer characterized using the Scanning Electron Microscopy (SEM) technique and the Atomic Force Microscope (AFM). The scanning electronic microscopy images showed nano-fibers with diameters at 43.3 nm while the AFM images show that the particle size distribution between 60 to 110 nm. The resultant polymeric composite materials were studied in different acidic and basic mediums. It was concluded that in the normal conditions, the hardness value was less than pure epoxy template and the hardness was increased with the increase of the epoxy-nanocellulose reinforcement ratio and the highest hardness of the template supported by 4 g of nanocellulose. The value of hardness is less for the polymeric compound (epoxy-nanocellulose resin) with increased immersion time in the acid and base solution. The values of hardness in the dry state before immersion in solutions, is greater that in the case of immersion in the acid and base solution where the values of hardness when immersion in the base solution are greater than their values when immersion in the acid solution.. The hardness values of the resultant polymeric composite were found to be lower in the acid and basic mediums. In addition, it shown that this effect was lower for basic mediums. Keywords: Polymer, Nanocellulose, Composite Materials, Epoxy, Resin.**RESUMEN** / **En este estudio, se sintetizó un compuesto de resina de epoxi-nanocelulosa de los residuos agrícolas (residuos de bagazo de caña de azúcar) disponibles localmente en Iraq. El polímero a nanoescala caracterizado por la técnica de microscopía electrónica de barrido (SEM) y el microscopio de fuerza atómica (AFM). Las imágenes de microscopía electrónica de barrido mostraron nano-fibras con diámetros a 43.3 nm, mientras que las imágenes AFM muestran que la distribución del tamaño de partícula entre 60 y 110 nm. Los materiales compuestos poliméricos resultantes se estudiaron en diferentes medios ácidos y básicos. Se concluyó que en condiciones normales, el valor de dureza era menor que la plantilla epoxi pura y la dureza se incrementó con el aumento de la relación de refuerzo de epoxi-nanocelulosa y la mayor dureza de la plantilla soportada por 4 g de nanocelulosa. El valor de la dureza es menor para el compuesto polimérico (resina epoxi-nanocelulosa) con un mayor tiempo de inmersión en la solución de ácido y base. Los valores de dureza en estado seco antes de la inmersión en soluciones son mayores que en el caso de la inmersión en la solución ácida y base donde los valores de dureza cuando se sumerge en la solución base son mayores que sus valores cuando se sumerge en la solución ácida. Se encontró que los valores de dureza del compuesto polimérico resultante eran más bajos en los medios ácidos y básicos. Además, demostró que este efecto fue menor para los medios básicos.**
Palabras clave: Polímero, Nanocelulosa, Materiales Compuestos, Epoxi, Resina.

1. Introduction

Composites materials produced using at least two constituent materials with altogether extraordinary physical or substance properties, which stay isolated and unmistakable inside the completed structure. Fundamentally, they ordered into two noteworthy sorts, i.e., auxiliary composites with extraordinary mechanical properties and utilitarian composites with different exceptional physical, compound or electrochemical properties. They utilized in a wide assortment of items, e.g., propelled rocket and flying machine segments, pontoon and scull bodies, outdoor supplies, sensor/actuator, impetuses and contamination handling materials, biomedical materials, and batteries, and so forth [1].

Nanocellulose is a nanoparticle made from cellulose and has many high physical properties such as strength, solidity, high viscosity and optical properties, in addition to mechanical properties. [2], high degree of crystallization [3]. It has also a high tensile strength and elasticity coefficient and is stronger than some materials such as Kevlar fiber and steel [4] .Nanocellulose is widely used in medical and pharmaceutical applications as well as for its uses in cosmetics [5] the nano cellulose possesses properties that improve the strength of its kinetic fiber bonding, making it an ideal material for strengthening compounds [6]. The main reason for the use of nanocellulose in composite materials is the great hardness of nanocrystalline cellulose as it uses nanostructures[7].Nanocellulose is used in decontamination as high adsorption substances such as the removal of oil stains in the sea [8] . It is also used in the textile and spinning industry and its improvement [9] and widely used in medical implants, engineering, tissue, wound healing, cardiovascular diseases [10]. The polymer is a macromolecule made up of small identical molecules that are bounded together by chemical bonds that often have covalent bonds. These molecules linked linearly in which they are called a linear polymer. Sometimes the polymeric molecule is called the branched polymer. The process of binding these simple molecules to polymerization is the polymerization process. Polymers are the number of repeating units in the polymer chain which have very high molecular weight[11] .Some polymers are natural such as wood, wool, cotton, rubber and they represent carbohydrates, proteins, nucleic acids and some polymers are industrial such as adhesives, building materials, paper, clothing, fibers, plastics … etc [12].

Epoxy resin is one of the most important types of heat-resistant resins and is made from the reaction of chlorhydrin with diphenol-propane [13] .The epoxy resin is used for covering the surfaces of the material. Its hardness, elasticity, adhesion and resistance to chemicals can characterize these sheets. They are formed by casting or applied to produce resistance tools with fiberglass. They have high mechanical properties and electrical insulation properties[14]. It used in the manufacturing of floorings and road coverings. It is can also be used in the manufacture of adhesives and materials for polyvinyl polymers. The transparent epoxy resin has a density of (1.10-1.25 g / cm3) and a glass transition rate of Tg (110 ° C) H z). [15].

In this work, we obtained nanocellulose successfully from locally available sugar cane residues. Nanosellulose application prepared by mixing polymeric complexes of nanosellulose with epoxy.

2. Materials and Methods

2.1 Materials

Sodium hydroxide and sodium hypochlorite were purchased from BDH, sulfuric acid was purchased from Sigma-Aldrich, epoxy resin and sulpheric acid was also purchased from Hanchel and hydrochloric acid was purchased from Baker Thomas.

2.2 Methods of Work

2.2.1 Preparation of Nanocellulose

The sugar cane residues were collected, washed and dried,. The bagasse which collected from the sugarcane plant in city of Amarah / Iraq, washed, dried treated with 1%NaOH to remove the xylene bleached, hydrolyzed using 35% sulfuric acid with strong stirring at temperature 30 ^{o}C and the suspension was then sonicated by ultrasounicator (sono-plus HD 2070) for 60 minutes[16]. Preparation steps summarized as shown in figure (1).

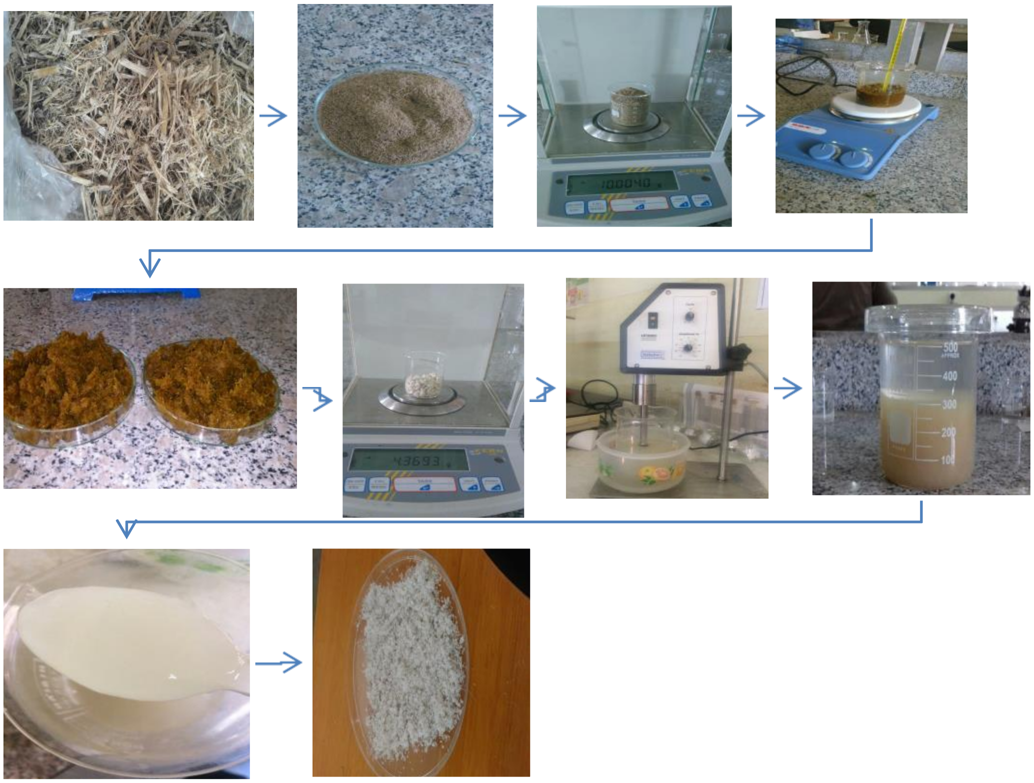

Figure (1) Preparation stages of nanocellulose from sugar cane residues

2.2.2 Nanocellulose Identification

Nanocellulose morphology was characterized using the Scanning microscopy (SEM) (model Quanta 200FEG) and the Atomic Force Microscopy (AFM) (model XE-100E) .

2.2.3 Preparation of polymer compositions

The templates were prepared by hand using epoxy resin in templates. The template was prepared manually using 0.6 cm thick glass plates and its dimensions (16 * 10 cm). The glass panels are stabilized by thermal wax. The template is prepared on a straight surface. The pure epoxy resin template is casted and added with ratio (1: 2). After that, the template of epoxy-nanocellulose compounds was casted.[17][18][19]

A quantity of epoxy resin is added and the quantity of the crucifixion in Baker is added and mixed with continuous and slow motion for 3 minutes for the purpose of homogenizing the mixture and eliminating the bubbles. Then, 2 g and 4 g of nanocellulose respectively added gradually with slow continuous stirring and then casting in the pre-prepared template. These templates stored at room temperature for 3 days. After that, these templates placed in oven for 5 hours at 50 ° C and left in oven until cooled. The templates then cut into small pieces (1 * 2 cm) for measurement of hardness of template by durometer.

Each block divided into 12 pieces. Each 5 pieces of the template immersed in two containers, one of which is 0.3N of NaOH. The other contains hydrochloric acid with the same concentration. The hardness measured once every 10 days for a whole month and the hardness measured before immersing the pieces in the solutions as shown in Figure (2).[20][21][22]

Figure (2). Stages of forming templates.

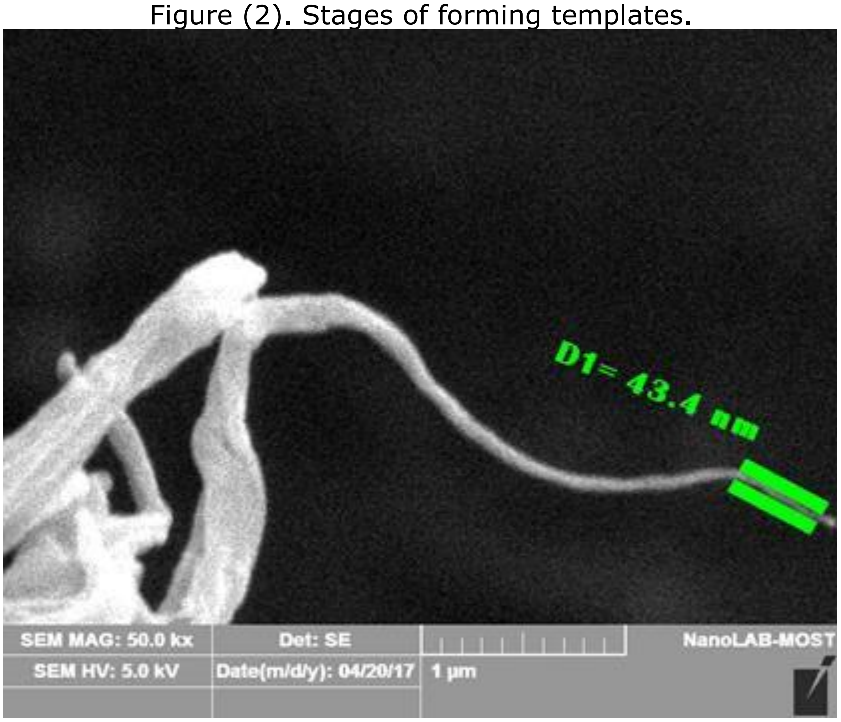

Figure (3). Scanning electronic microscopy of nanocellulose prepare from sugar cane residues

3. Results and discussion

3.1 Characterization of Nanocellulose.

Nanocellulose was characterized using the following techniques:

3.1.1 Scanning electron microscopy technique

The ultrasound treatment of the prepared sample of sugarcane residues for 60 minutes led to the production of nanocellulose in the form of nanofibers. The scanning electron microscopy technique was used in the characterization of the nanofibers of the prepared compound. The SEM image of prepared nanocellulose showed that nano fibers diameters are less than 50 nm as in figure (3).

3.1.2 Atomic Force Microscopy Technique

Atomic force microscopy technique was used to study the external appearance, diameter estimation and particle size distribution of the nanoparticles of the prepared nanocellulose. Figure (4) shows the image of the atomic force microscope of the prepared nanocellulose, where the image on the left is two dimensional and the image on the right is three dimensional. This image indicates that the prepared nanocellulose has a small particle size distribution.

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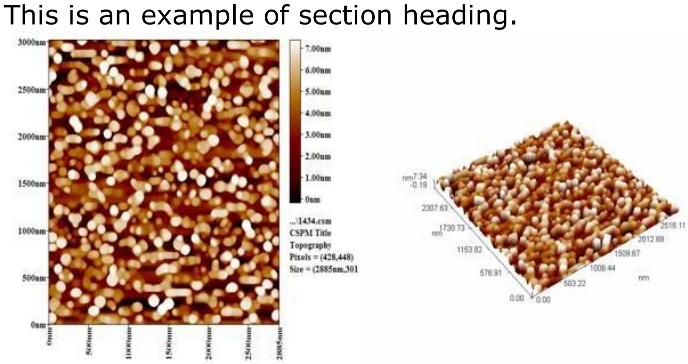

Figure (4). Image of Atomic Force Microscopy of Nanocellulose Prepared from Sugarcane Residues

The prepared nanocellulose particles show a very tiny particle size distribution as shown in Figure (5).

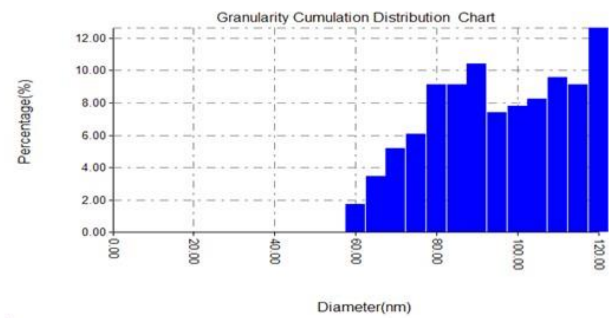

Figure (5). Average particle size distribution of nanocellulose produced from sugarcanes.

count distribution and diameters of nanocellulose are indicated in Table (1). The table shows that the count of nanoparticles obtained with diameters ranging from 75 nm to 165 nm.

Table (1) Count distribution of nanoparticles of prepared nanocellulose from sugarcane residues.

Diameter(nm)<	Volume(%)	Cumulation(%)	Diameter(nm)<	Volume(%)	Cumulation(%)	Diameter(nm)<	Volume(%)	Cumulation(%)
75	9.79	9.79	105	14.47	77.87	135	1.7	97.87
80	10.21	20	110	4.26	82.13	140	0.85	98.72
85	8.94	28.94	115	5.53	87.66	145	0.43	99.15
90	10.64	39.57	120	2.13	89.79	160	0.43	99.57
95	12.77	52.34	125	3.83	93.62	165	0.43	100
100	11.06	63.4	130	2.55	96.17			

4. Hardness Test of Prepared Composite Polymer

In this study, hardness tests was carried using durometer hardness instrument. it is concluded that in the normal conditions the hardness value was less than that of the first template (pure epoxy template) and the hardness increased with the increases of the epoxy-nanocellulose reinforcement ratio and the highest hardness of the template supported by 4 g of nanocellulose. As indicated in tables (2) and (3), the value of hardness is less for the polymeric compound (epoxy-nanocellulose resin) with increased immersion time in the acid and base solution. The values of hardness in the dry state before immersion in acid and base solutions, is greater than in the case of immersion in the acid and base solution where the values of hardness when immersed in the base solution are greater than their values when immersed in the acid solution.

The lowest values of hardness were recorded after immersion in the pure epoxy resin, which was submerged in the acid solution. Acid solution affects the hardness of the template more than the base solution because the acid works to weaken the material as the spread of the acid particles during the composite material that leads to weakening and breaking the bonds that lead to generating bubbles. The bubbles are regarded a phenomenon of deformation as the entry of molecules of acid into the sample leads to weak bonding between the components of the composite substance between the epoxy base material and the additive nanocellulose. This in turn increases the porosity, thus increases the absorption of acid, which works on increase the plasticity material, and thus lower the value of hardness as shown in the figs (6) and (7).

Table (2) Hardness values for polymer composites (epoxy resin + nanocellulose before immersion (B.I) and after immersion) in acidic solution.

No.	Sample Composition	Hardness (N/mm^2)			
		B.I	Immersion Time in acid (days)		
		0	10	20	30
1	Epoxy Pure	39.5	39	26.8	24.4
2	Epoxy + 2g nanocellulose	41.5	41.3	31.3	30.1
3	Epoxy + 4g nanocellulose	49.2	43.8	35.7	34.4

Figure (6). Changing in the hardness values of the templates immersed in the acidic solution

Table (3) .Hardness values for polymer composite (epoxy resin + nancoellulose before immersion (B.I) and after immersion) in basic solution.

No.	Sample Composition	Hardness (N/mm^2)			
		B.I	Immersion Time in acid (days)		
		0	10	20	30
1	Epoxy Pure	39.5	41.4	29.8	28
2	Epoxy + 2g nanocelloluse	41.5	40.4	34	32.2
3	Epoxy + 4g nanocelloluse	49.2	44.2	37.1	36.1

5. Conclusions

Nanocellulose successfully synthesized from locally available sugarcane residues. The SEM image of prepared nanocellulose showed that nano fibers diameters are less than 50 nm in average while AFM images show that the count of nanoparticles obtained with diameters ranging from 75 nm to 165 nm. Epoxy-nanocellulose composite was prepared by mixing epoxy resins with nanocellulose (2% and 4% nanocellulose). The hardness tests shows increase in value with the increases of the epoxy-nanocellulose reinforcement ratio and the highest hardness of the template supported by 4 g of nanocellulose. The hardness values of the composite epoxy-nanocellulose were found to be lower in the acid medium than the base medium.

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