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Synthesis of Composite from Bacterial Cellulose and Gold Nanoparticles

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Abstract. A process of bacterial cellulose gold nanocomposite has been investigated based on experimental work and cited literature. A literature review on the production process is carried out in this study. Bacterial cellulose is a high crystalline fabric material generally used in biomedical applications. A Nanocomposite was made by synthesis from gold and bacterial cellulose. The experimental work includes growing, and isolating bacterial cellulose, preparation of gold Nanoparticles and preparation of Nano composite. Nanoparticle's formation and adsorption on the cellulose tissue have been observed visually, where a colour change was observed. The predicted particle size for the gold nanoparticles was (2-100) nm.

1. Introduction

Bacterial cellulose (BC) was invented by Brown in 1886, he found a creation of material in a glucose solution created by a special kind of bacteria "Acetobacter xylinum" whose structure is similar to that of cellulose [1]. Chemical formula $(C_6H_{10}O_5)_n$. Nnaturally, the bacteria synthesize extracellular polysaccharides. The chemical structure is shown in Figure 1.

Figure 1. Chemical structure of Bacterial cellulose [2].

BC built from linear glucan molecules attached with hydrogen bonds seems similar to plant cellulose [3]. BC has been characterized by high porosity. BC is a nanomaterial that appears as a gel. Its particle size (10 -100 nm), so it has a capability for water retention [4]. (See Figure 2)

Drug-resistant bacterial infections have serious problems accompanied by wound healing. Bacterial cellulose decorated by organic carrying materials and modified gold nanoparticles were discovered as a dressing nanocomposite for treating bacterially infected wounds. Gold (AuNP) nanoparticles process

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unique optical, antibacterial, and catalytic properties, usually in situ synthesis of these NPs in BC for the fabrication of functional materials [5].

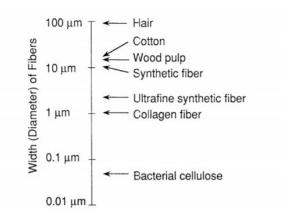


Figure 2. Sizes of synthetic and naturally occurring fibers [6].

1.1 BC Applications

Table 1. Applications of Bacterial Cellulose

Application	Example	Refs.
	-	
Biomedical	Wound dressing material, Wound healing, BC-based	[7] [8][9]
	materials for skin tissue repair dental implants	
cosmetology	Emulsion stabilizer.	[10]
Fabrics	Textiles.	[11]
Paper industry	Durable Papers.	[12]
Membranes	Ultrafiltration equipment	[13][14]
Food	Thickening and gelling, stabilizing, water-binding, and as a packing material	[15]
Acoustics	Membrane speaker	[16]
Science	Protein immobilization, culture medium	[17]
Catalysts	Catalyst support	[18] [19].

1.2 BC Preparation and Production

The production process faces many important restrictions like low yield, biochemical reaction time, the material of construction –etc. all of these are considered as reasons which limit the construction of industrial processing of BC when evaluated from an economic view. Many studies were conducted to overcome the limitations. Optimization, evaluation of substrate mass transfer on the biochemical reaction [20], and effect of reaction container wall on surface culture [21]. Research and development

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on reactors and processes continued. Cellulose production depends heavily on several factors such as the growth medium, environmental conditions, and the formation of byproducts. The best carbon sources in the fermentation medium which give the highest yield is glucose, followed by fructose, sucrose, and ethanol [22]. The problem with using glucose is the formation of gluconic acid side product, which lowers the pH of the culture and reduces the cellulose product, this problem was solved by the addition of (1%, w/v) lignosulfonate[23], or (2%) acetic acid [24]. Selection of reactor types should be conducted on pilot results. Several reactor types were reported for the biochemical fermentation reaction (see Table 2)

Reactor	C g/L	Time	Ref.
Classic reactors	1.69-2.5	12-20 day	[25]
Internal loop air lift reactor	3.8 - 8.7		[26][27]
Rotating disk reactor*1		84 h	[28][29]
Agar surface modified reactors	5.472	140h	[30]
Biofilm reactor	7.05	120h	[29]
STR +Spin filter*2	5.65	140h, pH 5	[31]

Table 2. Reactors used for Synthesize of Bacterial Cellulose

It's important to review preparation methods to assess the process design requirements and process flow diagram. For economic requirements, researchers aim is to reduce the cost of products. using Acetobacter xylinum. The challenge for minimizing cost through changing raw materials such as low-quality date syrup in Static batch fermentation method [25], or equipment design as shown in Table 2. It was reported for the process of cellulose formation from sugar. It was found that the production process was controlled by the diffusion of atmospheric oxygen [32].

1.3 Preparation of Nano Gold

Gold nanoparticles (AuNPs) have been widely used in biotechnology due to their unique properties and multiple surface functionalities. [33] [34] [35]. One of the synthetic methods for producing AuNPs, mixing (HAuCl₄) with citric acid in boiling water, where the citrate acts as both a reducing and stabilizing agent [36], particle size controlled by gold to citrate ratio [37].

This protocol has been used to synthase dilute solutions of moderately stable spherical AuNPs with diameters of (10 - 20 nm) [38].

Different methods for composites fabrication of gold nanoparticles, bacterial cellulose was studied. They looks similar in the principle, such all of them used reducing agents. They differ in number of steps or the type of reducing and linking agents like the one step poly-ethyleneimine as the reducing and linking agent with the addition of different halides [39]. Two steps amidoxime surface- functionalized bacterial cellulose [18]. Radical polymerization method was carried out using a monomer of 2-hydroxyethyl methacrylate to get a uniform distribution of the AuNPs on the membrane surface during the synthesis stage [18]. In situ polymerization of aniline by ammonium persulphate [40].

^{*1} Horizontal fermenter equipped with rotating discs or rollers rotated at 4 rpm.

^{*2} Fermentations are equipped with a turbine impeller and a spin filter.

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An invention related to drying of BC, called drying under tension, the method integrates the dehydrating and drying the BC produced in agitated culture followed by homogenization. The aim was to restore the properties of BC after drying [41].

Purification of BC can be done by boiling distilled water followed by soaking in NaOH solution for 24 hr. then rinsing with distilled water till reaching a pH of 7 [42].

2. Materials and Methods

2.1 Materials

- Gold (99.99%): local market
- Hydrochloric acid (37%)
- Nitric acid (68-70%)
- Butanedioate, 2-hydroxy-, sodium salt
- Iraqi mother vinegar was brought up from province of Diyala, east of Baghdad.
- Zahdi date, length of date piece (3.4 4 cm), diameter 2.4-2.5 cm, core form about 11% its weight, total sugars about 67%. Total dissolved materials 77.8%.

2.2 Methods

2.2.1 Preparation of Zahdi juice:

The date juice was prepared in a method that is similar to the procedure in the cited reference [43]. A 500gm date of cleaned and pitted date, cut into small pieces and leached with distilled water (2:1) at 70°C. The extracted juice was filtered and then concentrated to the required concentrations by evaporation at 70°C. The prepared date juice concentration of 40%.

2.2.2 Preparation of BC

Bacteria were put in water at (10% ~ 15%) concentrations for 10 days. A (2 - 3) drop of Ammonium acetate were added frequently every two days as an activator, the bacterial cellulose was mixed by magnetic stirrer and distributed into sterilized clean pots.

Vinegar bacteria was added with Two drops of ammonium acetate and the stirring continued for five minutes. Then containers were closed and sealed, incubated at a temperature of (30 C°). the cans were opened and fed by date syrup every 4 days frequently within two weeks period. Homogeneous bacterial cellulose was enlarged to three times its volume.

The bacterial cellulose colony was transferred to an open dish. Washed with distilled water three times then bleached by immersion in sodium hypochlorite solution bacterial, the resulted white colour BC was poured in distilled water.

2.2.3 Gold Nanoparticles:

0.5g Gold was melted in a mixture of (6ml HCl (0.1 M) + 2 ml HNO3 (0.1 M)) with boiling then dissolved in 500 ml to distilled water to give concentration (1000 g/L).

2.2.4 Preparation of gold Nanocomposite:

Chloroauric acid is prepared by dissolving 0.5 gm. of gold in aqua regia $(4HCl + HNO_3)$ $Au + HNO_3 + 4HCl \rightarrow HAuCl_4 + NO + 2H_2O$

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In the next step, 8.0 mL of the Chloroauric acid was added to a solution mixture of (10 ml sodium maleic + 10 ml sodium hydroxide) in a test tube fixed in a water bath near boiling point, after that, the mixture was subjected to further heating without stirring. Red colour was achieved after 5min at this stage a piece of bacterial cellulose was added. Colour changes continued after 10min. a brown hue was shown. After 15min. a dirty gray colour appears, and after 20min. aggregated particles were visible. (See Figure 3).

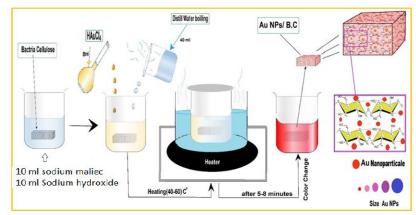


Figure 3. Preparation pieces of gold/bacterial cellulose nanocomposite.

3. Results and Discussion

Bacterial cellulose has high purity due to a lack of lignin and hemicellulose; this property makes bacterial cellulose. Bacterial cellulose is considered as a non-cytotoxic, non-genotoxic, and highly biocompatible material. Bacteria size increased after 10 days. It was observed that the anaerobic fermentation led to the growth of bacterial cellulose is observed as shown in Figure 4.

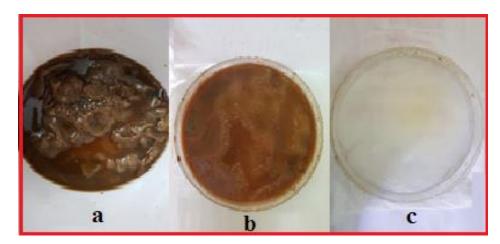


Figure 4. a- Mother of vinegar. b- after 21 days in an incubator at 30 °C. (c) Bleached bacterial cellulose.

The formation and adsorption of nanoparticles on the surface of cellulose tissue have been observed visually, a colour change was observed. The colour change is attributed to the Plasmon surface effects. Butanedioate, 2-hydroxy-, sodium salt used as reducing agent. (See Figure 5).

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Figure 5. Gold/bacterial cellulose nanocomposite.

Particle size for nanomaterials prophesied from absorbance. Moreover, wavelength values. The predicted value for the size of gold nanoparticles, 2-100 nm by maleic It is corresponds to the top absorbance value of 305-623 as shown in (Figure 6).

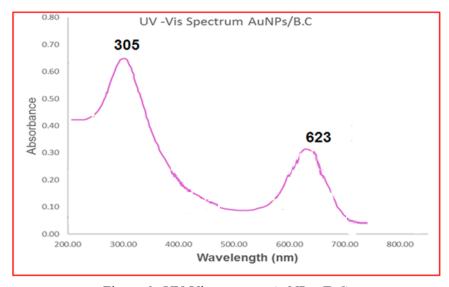


Figure 6. UV-Vis spectrum AuNPs. /B.C.

Morphology analyses illustrated in Figure 7, images show a cellulose tissue composed of porous fibers with a diameter of (0 - 70 nm). A scanning electron microscope explicates the structure of bacterial cellulose, ultrafine fibers are distributed in nano dimensions of infinite precision interrelated in a woven form that helps gold ions to spread in a spongy structure.

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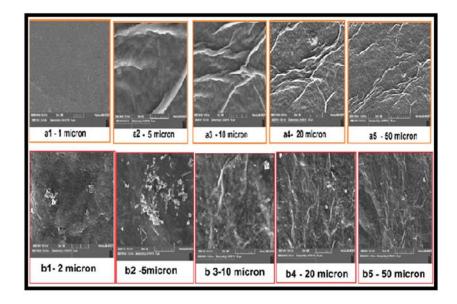


Figure 7. SEM micrographs of the a- pure bacterial cellulose b- by malic salt (Sodium2- hydroxy Butanedioate).

Atomic force microscope (AFM) Was used to examine the pure bacterial cellulose and the Au NPs/B.C. It used to predict pure cellulose and 3D as shown in Figure 8, it was being adsorbed on the bacterial cellulose tissue that sizes (47nm), (35nm). This indicates successful adsorption of gold nanoparticles on bacterial cellulose tissue.

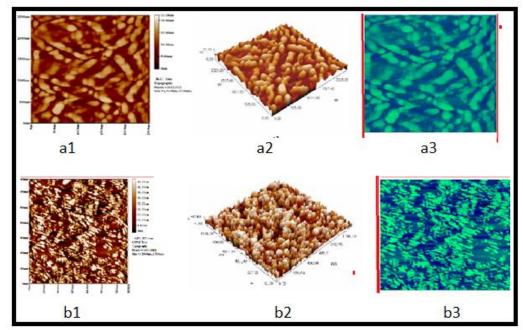


Figure 8. AFM image of bacterial cellulose. 1-2D 2-3D 3-photo film at 25 °C

3.1 Suggested process

The suggested process is for processing 1ton Zahdi date pilot plant for the production of gold nanoparticles - bacterial nano cellulose.

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Basis 1 Tons Zahdi date, Sugar 67%.\

Date syrup: for each 1 kg date, 2 L water required to produce syrup 22-25%bx Recovery of Sugar 60%. A preliminary outline of the suggested process is described by Figure 8-a for the date syrup process and Figure 8-b for bacterial cellulose- Au-NP.

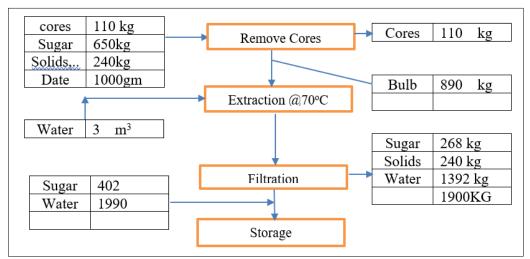


Figure 9(a). Schematic drawing for date syrup block diagram.

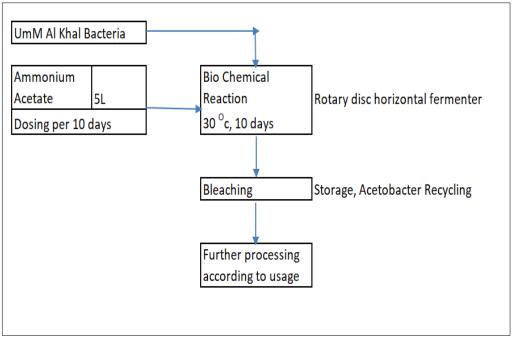


Figure 9(b). Schematic drawing for Au-NP Bacterial Cellulose block diagram.

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4. Conclusions

- Laboratory results have been proved that the Iraqi mother vinegar is a good bacteria source for synthase of bacterial cellulose.
- Successful adsorption of gold nanoparticles on bacterial cellulose tissue.
- Based on morphology analyses, it is concluded that cellulose tissue is composed of porous fibers with a diameter of 0 to 70 nm size.
- This method gives extra pure nano bacterial cellulose with good mechanical properties

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