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Synthesis and Thermal Stability studies of Nanocomposite Cotton Fabrics (NCCF) with *In situ* generation of CuNPs by Bioreduction Method

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Abstract: *Using fresh green Citrus lemon leaf extract, as a bio-reductant, infused into pristine cotton fabric resulted in cotton matrix. The nanocompostie cotton fabric systems have been synthesized by immersing cotton matrix into different concentrations aq. $CuSO_4.5H_2O$ Source solutions. The Copper Nanoparticles (CuNPs) were in situ formed in cotton fabrics due to bioreduction by infused phytochemicals in the cotton matrix. This Nanocomposite Cotton Fabric (NCCFs) @ in situ formed CuNPs were analyzed with XRD and thermal stability by TGA.*

Key words*: Citrus lemon leaf, Cotton fabrics, Au and Cu nanoparticles, In situ formation, TGA*

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I. Introduction :

In order to minimize environmental impact and applications of Metal Nanoparticles (MNPs) in health sector, essentially depends on non usage of toxic or harmful chemicals during their synthesis. Many areas of research including medicine, catalysis, photo electricity and industrial manufacture etc., have been revolutionized by nanotechnology. The demand for the development of materials with multifunctional properties has driven different studies encompassing Metal Nanoparticles (MNPs) and Cotton fabrics. This paper has been focusing on Synthesis and studies of Thermo gravimetric analysis (TGA) of cotton fabrics with in situ formed Cu nanoparticles through green synthesis methods where reducing and stabilizing agents are obtained from lemon leaf extract.

II. Materials And Methods

Materials

The $CuSO_4.5H_2O$ is procured from *SD fine* Chem., was used as received without further purification. The pristine Cotton fabrics with white color were procured locally (Hyderabad, India). These fabrics were initially washed in deionised water and then dried in the air.

Leaf extract Preparation

Citrus lemon belongs to *Rutaceae* which is widely grown commercially worldwide. The fresh green Citrus lemon leaves were collected from campus of *Science and Humanities Block of University College of Engineering, Osmania University*, Hyderabad, India. After thoroughly washed using deionised water and dried in shade. A quantity of 100 grams of the dry leaves was soaked in 900 ml of preheated deionised water (80˚C) by water bath method, for 20 minutes duration. After cooling to ambient temperature, extract was filtered which may be readily usable or can be stored at 5 ^oC for 3 to 4 days for further usage. The lemon leaf extract as plant extract contains phytochemicals consisting of active alkaloids, phenols, terpenoids, quinines, amides, flavonoids, proteins and alcohols etc.[1].

Cotton Matrix Preparation

The pristine cotton fabrics had been procured from local market. The thoroughly washed pristine cotton fabrics of dimensions of 150mm ×100mm were immersed along the inside walls of glass beakers which contain prepared lemon leaf extract. These systems were then placed on magnetic stirrer at room temperature and maintain stirring rate 300rpm for 24 hours. A thin phytochemical layer of citrus lemon leaf extract could be adhered strongly by molecular forces on cotton surface (2). The cotton fabrics colour turned in to light greenish

during this process indicating the infusion of the phytochemicals of lemon leaf extract. The Cotton Fabrics infused by lemon leaf extract were employed as Cotton Matrix.

Nanocomposite cotton fabric (NCCF) with *In situ* generation of AuNPs :

The system of aq. $CuSO4.5H_2O$ solutions with different concentrations i.e., 1mM, 5mM, 25 mM, 125 mM and 250mM were prepared in beakers separately. In each, beaker a Cotton Matrix piece was introduced. These systems were then placed on magnetic stirrer at room temperature and maintain stirring rate 300rpm for 24 hours. During this process, the cotton matrix colour turned from light greenish to brown, primarily which may be due to Cu particles deposition on the cotton fabric which is designated as Nanocomposite Cotton Fabric (NCCF). In the NCCF, lemon leaf extract reduced CuNPs were absorbed and diffused on the cotton fabric surface for electrostatic interaction between the Cu ion particles and negatively charged –OH groups in the cellulosic chain of cotton (3). The NCCFs having in situ formed CuNPs were thoroughly washed in deionised water and dried. The NCCFs colour was retained even after repeated washings.

Characterization and Results :

i. Digital Images of NCCF @ CuNPs:

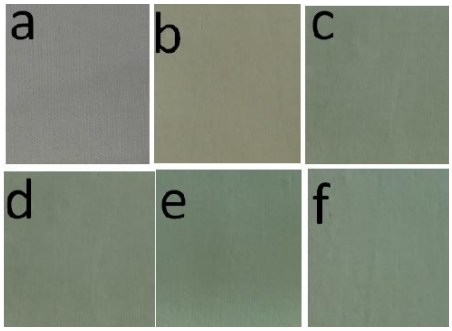

Fig 1 . Digital images of matrix (a), NCCFs made using 1 mM (b), 5 mM (c), 25 mM (d), 125 mM (e) and 250 mM (f) source solutions

In order to visualize the changes between the pristine white cotton fabric, cotton matrix and the nanocomposite cotton fabrics (NCCFs) with in situ generated CuNPs, using 1 mM, 5 mM, 25 mM, 125 mM and 250 mM source solution concentrations, their digital images were recorded by Sony digital Camera and are presented in Fig.1 a, b, c, d, e and f respectively. The color of the matrix changed to light brown due to insitu generated CuNPs by bioreduction of infused lemon leaf extract of cotton matrix.

ii. XRD Analysis

In order to confirm and study the effect of the generated CuNPs on the crystalline nature of the NCCFs, X-ray analysis was carried out. The X-ray diffractograms of the matrix and the NCCFs with generated CuNPs are shown in Fig. 2.

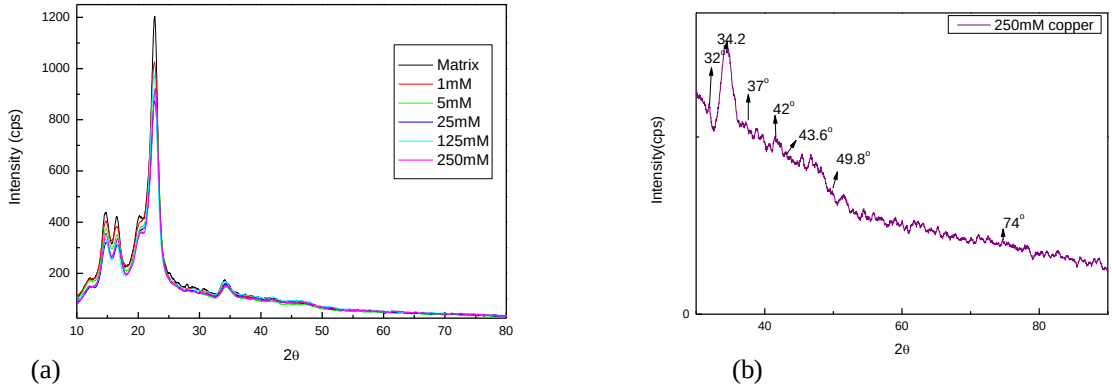

Fig. 2 X-ray diffractograms of the matrix and the NCCFs with in situ generated CuNPs using 1 mM, 5 mM, 25 mM, 125 mM and 250 mM source solutions (a) and Expanded diffractogram of the NCCF with in situ generated CuNPs using 250 mM source solution in $2\theta = 35^o$ to 80^o (b).

It can also be observed that the intensity of the main peaks of the NCCFs is lower than that of the matrix indicating that the generated CuNPs in the NCCFs lowered the crystalanity of the NCCFs [4] .

From Fig. 2, it can be seen that the diffractograms of the matrix and the NCCFs with in situ generated CuNPs are almost at the same 2θ positions. The main common peaks in the matrix and the NCCFs at 2θ= 14.6°, 16.5° and 22.6° arose due to the reflections from (1-10), (110) and (200) planes of cellulose-I structure [5]. The other weak peak at 2θ=34° arose due to the reflections from (004) plane of cellulose [6]. In addition to these main peaks, there exist very faint peaks which are obscured by the intense main peaks. In order to observe these masked peaks, the diffractogram of the NCCFs with in situ generated CuNPs using 250 mM source solution was expanded from2θ= 35° to 80° and presented in Fig. 2 (b). From Fig. 2 (b), the peaks observed at 2θ=42°, 49.8° and 74° correspond to the reflections from (111), (200) and (220) of CuNPs [7]. Besides these, the bands observed at 2θ=37°and 61.3° belong to the reflection from (111) and (220) planes of Cu_2O nanoparticles [8]. Thus the NCCFs had both CuNPs and Cu_2O NPs. This is understandable as some of the CuNPs were oxidized to Cu_2O NPs and Cu is a good oxidizing agent [9].

iii. Thermo gravimetric Analysis :

a. NCCF @ in situ formed CuNPs

In order to study the effect of the generated CuNPs on the thermal stability of the NCCFs, the thermo gravimetric analysis was carried out. The derivative thermograms of the matrix and the NCCFs using 1 mM, 5 mM, 25 mM, 125 mM and 250 mM source solutions are presented in Fig. 3

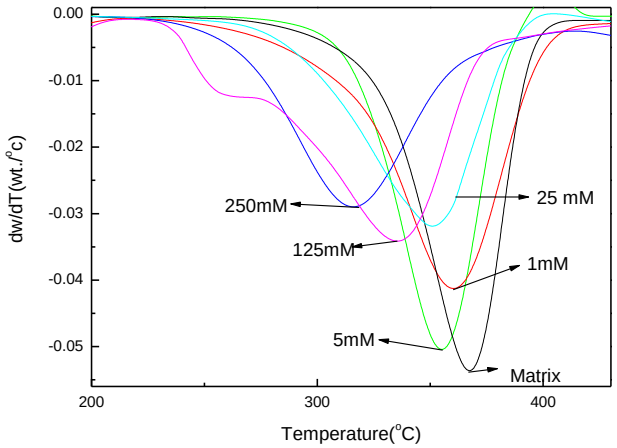

Fig.3 Derivative thermograms of matrix and the NCCFs prepared using 1 mM, 5 mM, 25 mM, 125 mM and 250 mM source solutions

From Fig.3, it can be seen that the inflection temperatures, i.e., temperatures where the degradation rate is maximum, of the matrix and the NCCFs with in situ generated CuNPs using 1 mM, 5 mM, 25 mM, 125 mM and 250 mM source solutions were found to be 368 °C, 361 °C, 355 °C, 349 °C, 336 °C and 316 °C respectively. This clearly indicates that the thermal stability of the NCCFs was lowered by the generated CuNPs. This may be due to the lowering of the crystalanity of the NCCFs by the generated CuNPs as revealed by the X-ray analysis. Similar observation was made in the case of [4].

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