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Preparation and Characterization of Iron Oxide Nanoparticles-Reduced Graphene Oxide Composites (IO-RGO) and Studying Some of its Medical Applications

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Abstract: A method of preparing iron oxide nanoparticles is green synthesis, in which pomegranate syrup extract is used in the synistation of the nanoparticles. In extracts of fruit quantities of pomegranate syrup, there may be some phytochemical substances such as polyphenols, flavonoids and organic acids that may act both as reducing and stabilizing reagents for the preparation of nanoparticles. Iron oxide nanoparticles was synthesized in the present research work by Sol – Gel auto-combustion technique some of these approaches and chemicals used are almost green and cost effective than the other methods of synthesis self combustion process has been ignited by natural extract of pomegranate Iraqi plant. and then to composite with reduced graphene oxide. Characterization of the prepared powder compositions was done throughPowder X-ray Diffraction [XRD] and Scanning Electron Microscopy [SEM].

Key points: Reduced Graphene Oxide (RGO), Graphene Oxide (GO), Oxide-Iron nanoparticles.

1-Introduction

The synthesis of nanoparticles has emerged as an important research area in the recent past because of the difference in properties that materials exhibit when compared in the nanoscale. All the above Fe-based nanoparticles showed great potential future application in various fields such as environment, medicine and in catalysis with the particular emphasis on iron oxide nanoparticles. Natural sources such as extracts serve as stabilizers and reducers of the oxidation processes in green methods of synthesis that are identified as more environmental friendly than traditional methods[1].

The synthesis of nanoparticles has gained much prominence recently since material properties differ remarkably at nano partition level. For instance, iron oxide nanoparticles have been considered for use in medical treatment, as well as in the enhancement of catalytic processes used in environmental remediation and other industries. Biological materials are used as reducing and stabilizing agents in green synthesis methods, which are now less harmful to the environment as compared to traditional methodology[2]

In this work, pomegranate syrup extracts are used as stabilizing and reducing agents in the green manufacture of iron oxide nanoparticles. Using pomegranate syrup improves the biocompatibility of the final nanoparticles while also offering a sustainable and economical method. The green synthesis approach is in line with the increasing need for environmentally friendly methods of producing nanoparticles.[3]

Experimental

2-1 Iron-Oxide Nanoparticle Synthesis

Bring a large number of pomegranates. Squeeze the seeds by hand to extract as much as possible after peeling the fruits. To get rid of any contaminants, strain and filter the juice with filter paper. Ferric chloride (FeCl3), 2.5 grams, should dissolve in 200 milliliters of purified water. Until the mixture dissolves completely, use a magnetic stirrer and keep the temperature at 80 degrees Celsius. Gradually add 50 ml of pomegranate juice extract to the previously made solution in a beaker, stirring constantly and keeping the mixture at 80 degrees Celsius. Dissolve 0.4 g of sodium hydroxide in 100 ml to get a solution with a concentration of 0.1 N. Stir the mixture continuously after adding the prepared sodium hydroxide solution. A gel that is reddish-brown forms. After letting the mixture settle for the entire night, filter the precipitate with a Buchner funnel to gather it. The final product should be dried in an oven set at 80 degrees Celsius. The precipitate from the previous step should be burned for three hours at 400 degrees in a muffle furnace.[4]

2-2 Preparation of Nanoparticles RGO

In a 500 milliliter reaction flask submerged in an ice bath, 1 gram of graphite, 1.5 grams of sodium nitrate, and 46 milliliters of sulfuric acid were fully combined and aggressively agitated at 0˚C for 15 minutes. After that, the solution was slowly mixed with 6 grams of potassium permanganate and allowed to cool for 30 minutes. For two hours at 35˚C, the resultant suspended solution was constantly swirled., during which 46 milliliters of water were slowly added to the suspension over 10 minutes, raising the temperature to 98˚C. Twenty more minutes were used for stirring the solution. The suspension was then agitated for ten minutes and diluted with 140 milliliters of warm water. Afterwards, after letting the mixture cool to room temperature, 215 milliliters of 30% H2O2 were added to the mixture to convert any leftover permanganate into soluble manganese ions. Ultimately, centrifugation was used to filter the resultant suspension, and distilled water and 10% HCl were used to wash it, then dried in a vacuum oven at 70˚C for 24 hours to yield 100 milligrams of graphene oxide (GO) [5]. The GO was dispersed in 1 milliliter of 37% HCl. Subsequently, 1 milliliter of 80% hydrazine hydrate was added, and the mixture had been heated for 2 hours at 95˚C. The reduced graphene oxide (RGO) was collected by filtration, repeatedly washed with water to get rid of extra hydrazine, and then dried for 12 hours at 100˚C in a vacuum oven.[6]

2-3 Preparation of iron oxide nanoparticles-reduced graphene oxide (IO-RGO)

Afterward, the RGO-Fe composite was prepared by sonication of 0.2 grams of RGO in 30 milliliters of deionized water along with a solution of iron oxide powders. The resulting mixture was stirred at 70°C for 15 minutes. Subsequently, 2 milliliters of ammonium hydroxide were added, and the mixture was stirred at 70°C for an additional 4 hours. Centrifugation was used to separate the final product, which was then cleaned with deionized water and dried.[7]

3. Results and Discussion

Nanoparticles of iron oxide were subjected to diffraction of X-rays (XRD) examination, as illustrated in Figure (1), in order to identify the diffraction surfaces of the nanoparticle crystal at diffraction angles of 33.2˚, 35.6˚, and 40˚. The diffraction angles match the formula for Fe3O4.[8] A closer look at the synthetic iron oxide nanoparticles SEM picture shown in Figure (2) showed that the particles were almost spherical in size and were closely packed.

A powder sample of GO underwent examination using an X-ray diffractometer, and its XRD pattern is shown in Figure (3). The pattern revealed a prominent diffraction peak at 11.84˚, This is equivalent to a d-spacing of 0.74 nm, which is higher than graphite's 0.33 nm. The addition of functional groups (epoxy, carboxylic acid, carbonyl, and hydroxyl) to the basal and edge planes of graphene oxide (GO) sheets is shown in Figure (4). Furthermore, the oxidation of graphite flakes (GT) is indicated by an increase in d-spacing. The traditional Debye-Scherrer equation was used to determine the thickness of GO sheets (t). where k, which is the Scherrer constant based on the size distribution and shape of the crystal (shape factor), λ is the wavelength (0.1541 nm) of the Cu Kα

radiation source, and β is the angular peak (FWHM) intensity in radian. where θ is the degree-wise Bragg angle. The 10-80° range was used to record the diffractograms, 5.34 nm in estimated thickness. The following relation can be used to estimate the number of stacked layers (n).[9]

$n = t/d$

It was discovered that there were (7) perpendicularly stacked GO sheets, where d denotes the interlayer gap.

The XRD patterns of RGO, as depicted in Figure (5), reveal a broad diffraction peak appearing at 2θ 24.63°, demonstrating a lackluster arrangement of the sheets in the direction of stacking. This suggests that the sample primarily consists of a few layers of RGO, has a d-spacing between layers of 0.36 nm, smaller than GO. both the GO and RGO morphology SEM images corresponding to the (002) planes of RGO, is illustrated in Figures (6)(7), respectively.

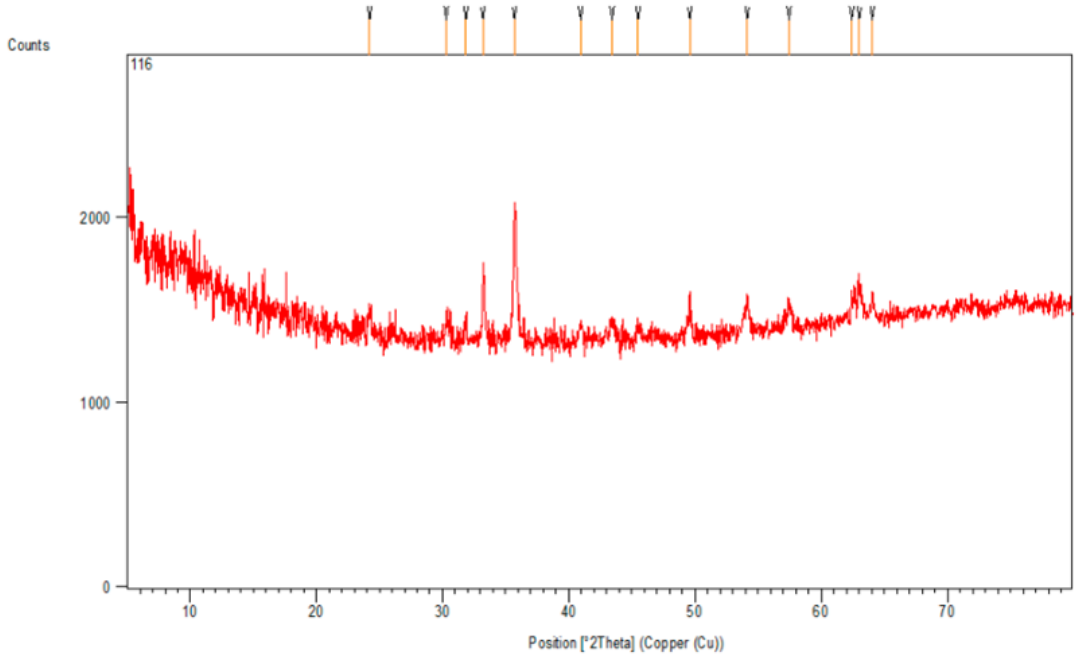

Figure 1: X-ray diffraction of iron oxide nanoparticles

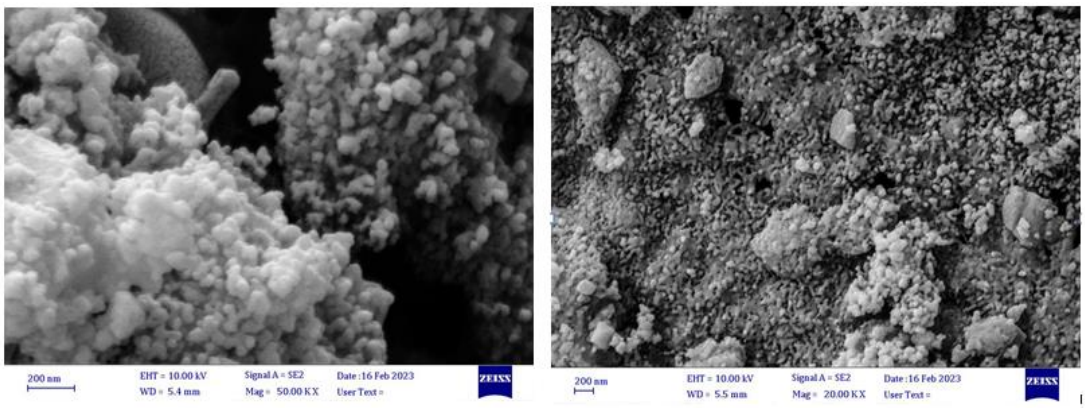

Figure 2: Synthesized Iron Oxide Nanoparticles in SEM Image

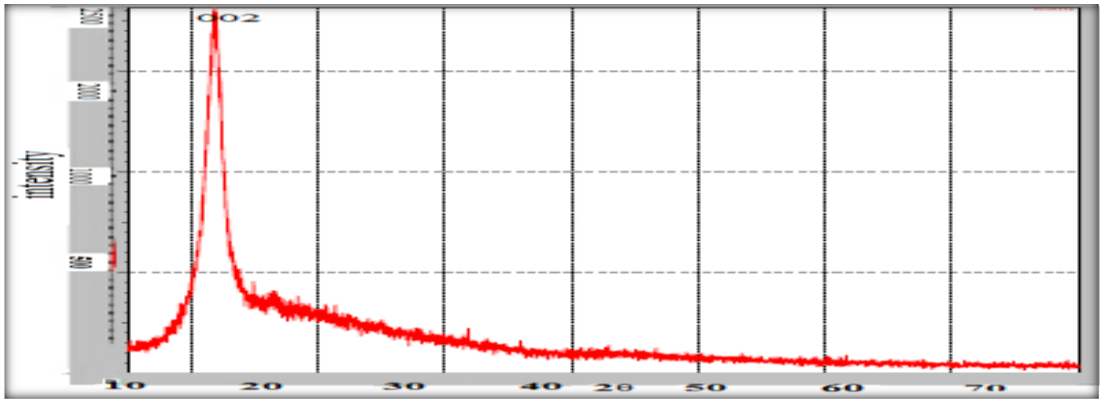

Figure 3. shows GO XRD patterns

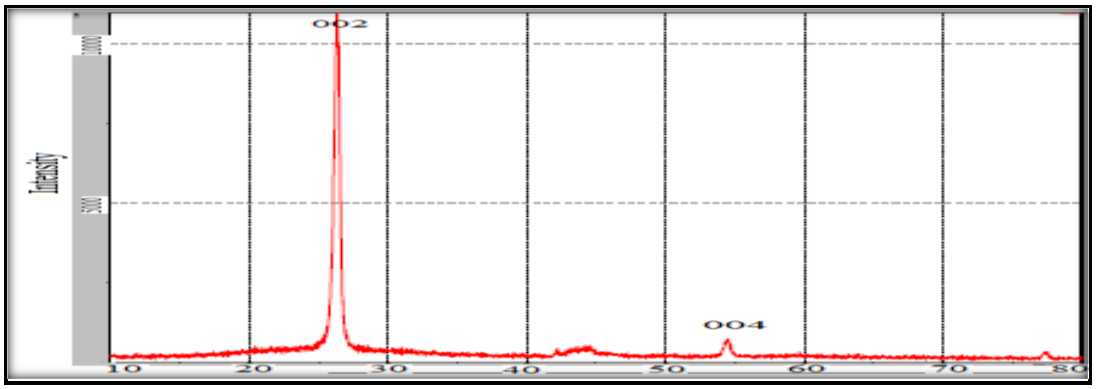

Figure (4) shows Graphite XRD patterns

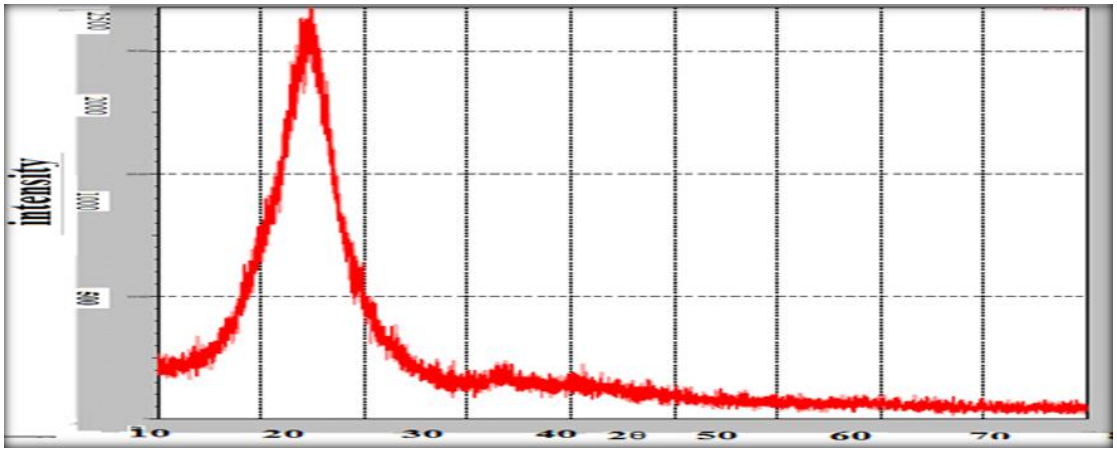

Figure (5) shows RGO XRD patterns

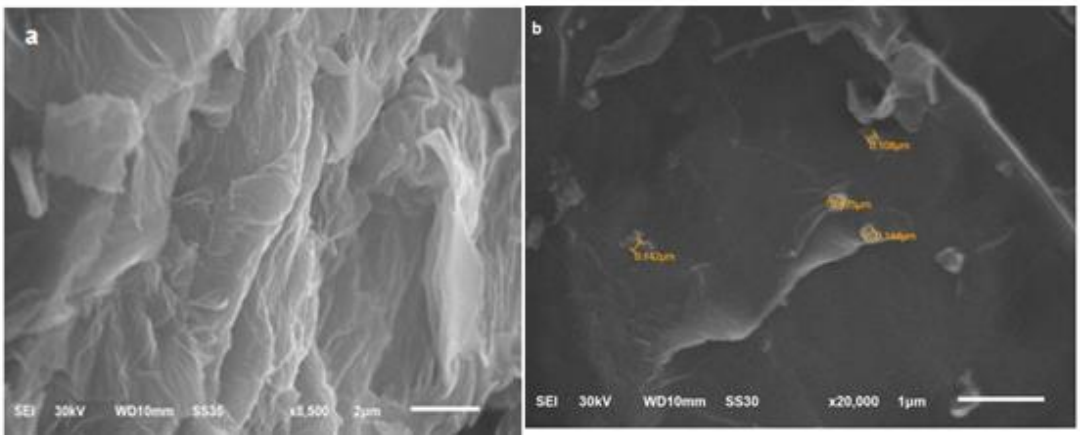

Figure (6 a,b): Synthesized GO in SEM Image

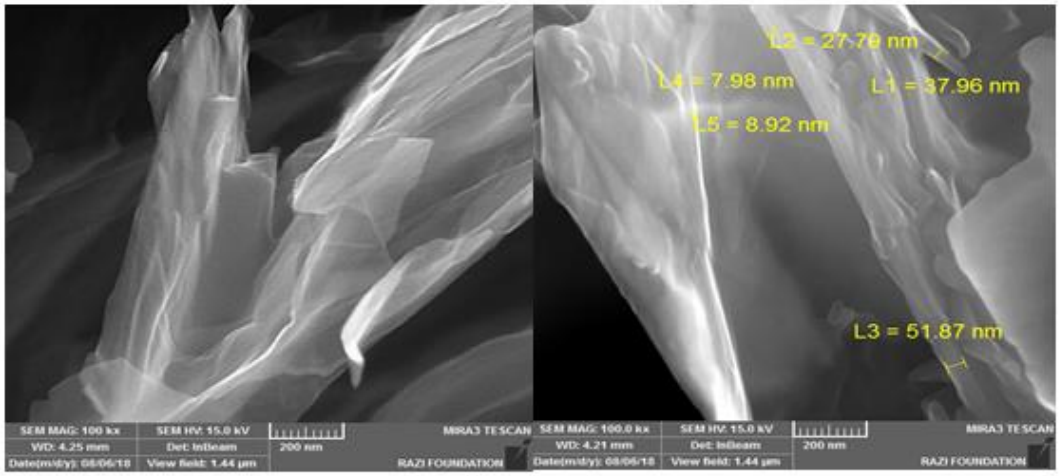

Figure (7): Synthesized RGO in SEM Image

The IO-RGO XRD patterns displayed in Fig. 8 (2θ 26.51o, 25.96o, 54.60). The RGO-IO's SEM picture is shown in Fig. (9)

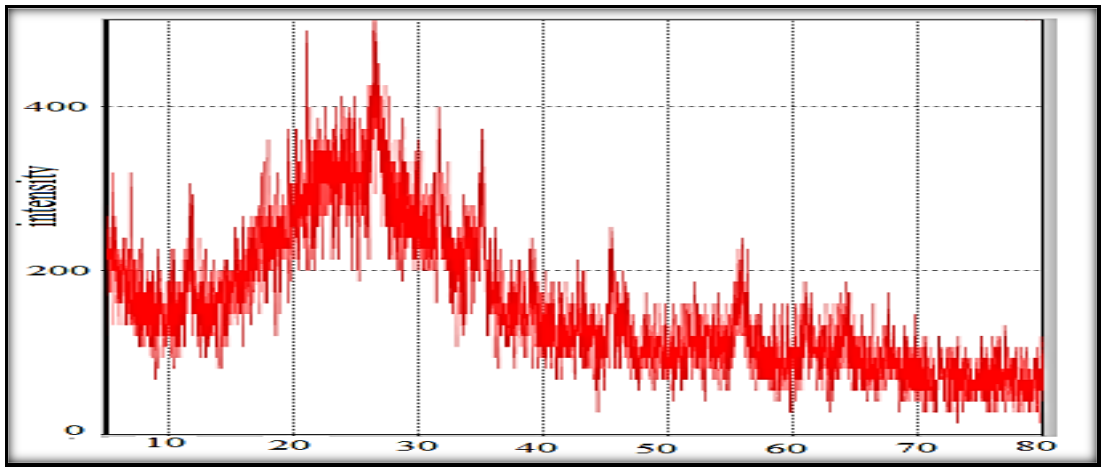

Figure (8) shows (IO-RGO) XRD patterns

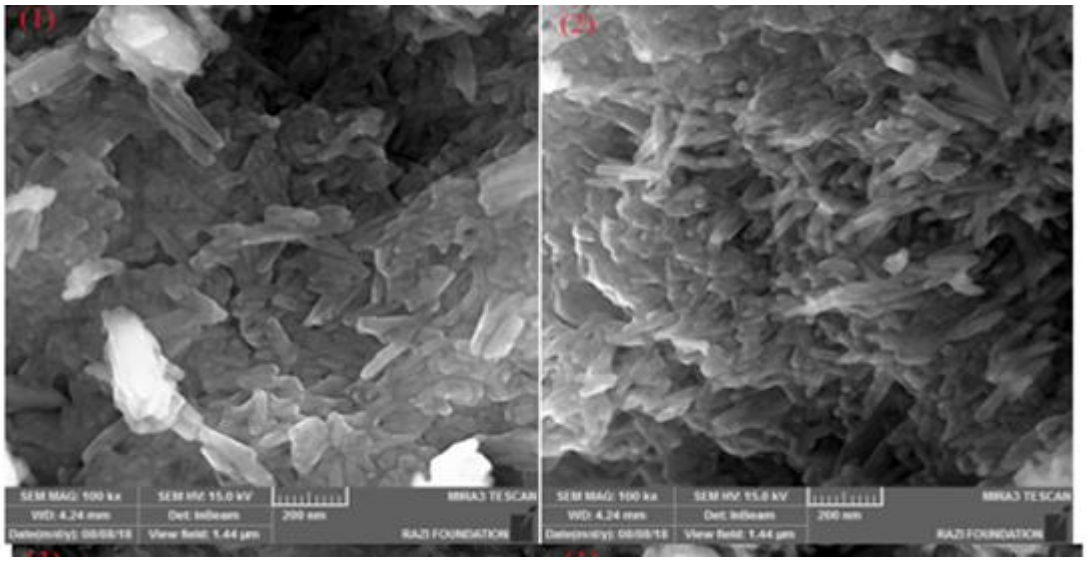

Figure (9): Synthesized RGO-IO in SEM Image

Table 1 and Figure 10 exhibit the antibacterial activity of the GRO-iron oxide nanoparticle assessed against harmful microorganisms.

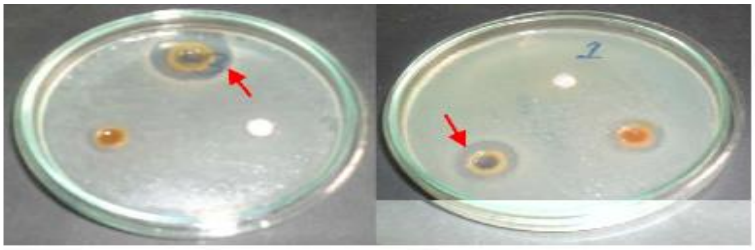

Figure (10): Antibacterial activity study

Table 1: (IO-RGO) nanoparticle antibacterial activity

Compound	*Staphylococcus aureus*	*Shigella flexneri*	*Bacillus licheniformis*	*Bacillus brevis*	*Vibrio cholerae*
RGO-IO	12 ± 0.35	0 ± 0.0	21 ± 0.70	10± 0.15	9 ± 0.0

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