Synthesis and Characterization of a Magnetised Nano Catalyst Derived from Pomegranate Seed Biomass

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Abstract. This study describes the synthesis and characterisation of a novel magnetically recoverable nano-catalyst (MAC) from pomegranate seed biomass, promoting value addition to agricultural waste for sustainable materials. The synthesis used a controlled pyrolysis and acid activation approach to prepare a porous structure of activated carbon support, followed by iron doping for magnetic properties and modifying surface properties. The structural and morphological properties of the prepared material were systematically studied. Fourier Transform Infrared Spectroscopy (FTIR) characterisation confirmed successful surface functionalization and metal-oxygen vibrations. X-Ray Diffraction (XRD) showed that crystalline iron oxide phases were encapsulated within the amorphous carbon for magnetic separation. The resultant magnetic nano-catalyst was found to be green, cost-effective, and recoverable for different heterogeneous catalytic applications in the spirit of sustainable material engineering.

Keywords: Magnetic nano-catalyst; Biomass-derived carbon; Sustainable materials; Heterogeneous catalysis.

1 Introduction

Fast industrialisation and urbanisation are fuelling high global energy demand, against a backdrop of continuously increasing pressure on conventional energy sources. Despite growing interest in renewable energy, fossil fuels still contribute the majority of global primary energy consumption. [2], yet their finite reserves and associated environmental issues [1] highlight an urgent need for cleaner, sustainable options. In this respect, eco-materials and green catalytic systems have become important areas of research in sustainable chemical engineering [3].

Catalysts are widely used in green chemistry to improve reaction efficiency and reduce waste generation. but even highly active homogeneous catalysts often create difficulties during practical use because their separation from the reaction mixture is tedious, their recyclability is poor, and they can impose additional environmental concerns [4]. Heterogeneous catalysts address many of these issues since they are generally more stable and can be separated easily after the reaction. However, several reported heterogeneous systems still rely on costly precursor materials or require multi- step and time-consuming synthesis routes, which reduces their feasibility for large- scale industrial applications.

To address these challenges, increasing attention is being directed towards the valorisation of biomass—converting agricultural residues into high-value functional materials. Pomegranate seed (PS), an abundant agro-waste, is a potential alternative to

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carbonaceous precursors for the synthesis of activated carbon (AC) with tunable porosity and a high specific surface area. Such carbon materials serve as excellent supports for heterogeneous catalysts. Introducing magnetic properties into heterogeneous catalysts allows simple separation using an external magnet, improving catalyst recovery.[6]. High surface area-to-volume ratios are provided by magnetic nano-catalysts [5], which also have the added advantage of being magnetically recoverable, making it simple to separate them from the reaction mixture using a magnet from the outside. This characteristic increases catalyst reusability, facilitates downstream processing, and improves overall process sustainability.
In this study, iron-doped magnetic activated carbon was prepared from pomegranate seed waste and characterised using FTIR and XRD. The goal is to synthesise an eco-friendly, inexpensive magnetically recoverable nano-catalyst from agricultural waste. FTIR and XRD were applied to characterise surface functional groups, crystalline phases and morphology. The results reveal the promising possibility as a solid and recyclable heterogeneous catalyst for various environmentally benign industrial processes.

2 Materials and Methods

2.1 Materials

Pomegranate seeds were obtained from the local market in Gandhinagar, Gujarat, India. Hydrochloric acid, sulfuric acid (5% normal), iron (III) chloride anhydrous, sodium hydroxide pellets (low chloride), cetyltrimethylammonium bromide (CTAB), and iron (II) sulfate heptahydrate were used in this study. All chemicals were obtained from institutional laboratory facilities and were of analytical grade.

2.2 Synthesis of Activated Carbon

Pomegranate seeds that could be found locally were used in the study as a raw material to obtain activated carbon. Before being dried at room temperature, 50 grams of fresh pomegranate seeds were processed using a laboratory dryer. Seeds were dried till the moisture content was less than 5% v/v. Particles ranging in size from 75 to 200 micrometres were created by grinding and shredding the dried seeds [7].
The crushed seeds were pretreated with hydrochloric acid (HCl) for an extended period of 24 hours to improve the porosity of the material and eliminate impurities. Following acid treatment, the material was thoroughly cleaned with distilled water until its pH was neutral. The seeds were cleaned and dried at room temperature [8].
To remove moisture content, the material underwent atmospheric drying before heating as part of the pretreatment process. Carbonisation processes were carried out in a muffle furnace to convert organic biomass into activated carbon. The materials underwent a preliminary heating process at 200 °C for 30 minutes to start the carbonisation process. To enable full carbonisation of the material, the furnace's heating process was prolonged from 250 °C to 300 °C over 4 hours of continuous activation [9].
This controlled thermal decomposition under limited oxygen conditions facilitated the development of a porous carbon structure while minimising combustion losses. To

prevent oxidising breakdown, the carbonised product was allowed to reach room temperature while the furnace switched to cooling mode, and the system remained sealed.

The obtained activated carbon was acid-washed with H_2SO_4 (5% v/v) to cleanse and activate the carbon pores. The mixture was kept in a closed vessel to avoid contamination overnight. This step helped in activating the prepared carbon due to the high acid content. The carbon was then washed with distilled water continuously until a neutral pH was obtained. Upon neutralising, the carbon was subjected to an oven for 4 hours or until the moisture content was less than 2% to obtain activated carbon [7].

2.3 Synthesis of Magnetised Activated Carbon (MAC) via Iron Doping

As precursor reagents, Iron (III) chloride ($FeCl_3$) and Iron (II) sulfate ($FeSO_4$) were used to create the magnetic nanoparticles. The aqueous solution was prepared by dissolving salts in 250 mL of distilled water and simultaneously being stirred to obtain a uniform solution. The $FeCl_3$ solution was first added to the reaction vessel containing 250 mL of distilled water and stirred using a magnetic stirrer. The $FeSO_4$ solution was then added gradually and was constantly stirred during this process to keep the system uniform. Sodium hydroxide (NaOH) Pellets were dissolved in 100 mL of distilled water and added dropwise to the reaction mixture to promote alkalinity and induce co-precipitation, which in turn facilitated the formation of iron oxide particles [10]. The reaction medium was supplemented with the activated carbon that had been produced earlier. At this point, a stabilising agent and surfactant, CTAB, were added to maximise the interaction between the carbon support and iron precursors while also improving their dispersion. The reaction mixture was continuously stirred for eight hours at 65–70 °C to fully synthesise the biocatalyst doped with magnetic material [11]. Following the reaction, the product was separated and allowed to dry at room temperature for 4 hours. At the end of this process, the iron-doped magnetic carbon was obtained, which was used as the catalyst for further research.

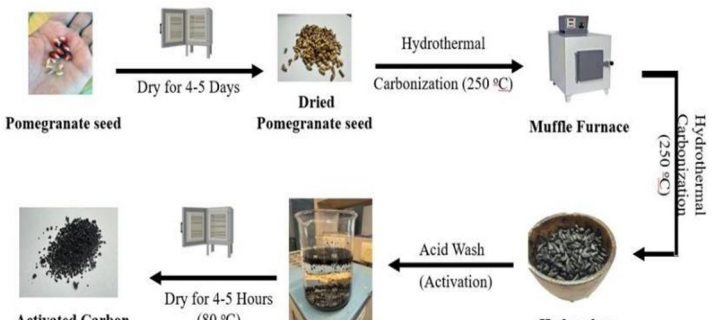

Fig. 1. Methodology for Activated Carbon synthesis from pomegranate seed

3 Results and Discussion

3.1 Fourier Transform Infrared (FTIR) Spectroscopic Analysis for Catalyst

FTIR analysis was carried out to identify surface functional groups present on activated carbon and iron-doped samples. The latter is expected to work as a heterogeneous catalyst in the preparation of biodiesel, while the former may act as a heterogeneous catalyst when doped with iron compounds in the process of producing biodiesel. FTIR spectroscopy senses molecular vibrations by means of absorption bands, which can be used to identify bonding and surface properties. Comparison of the FTIR spectra revealed the differences in transmittance between the two samples. The red and black are the magnetised and precursor-activated carbon spectra. In the typical absorption region associated with metal–oxygen (Fe–O) stretching vibrations, a notable change is observed [12]. This band (below 700 cm^{-1} in the MAC spectrum) is absent (or much less intense) in the AC spectrum, indicating the inclusion of iron species in the carbonaceous structure. Hydroxyl and carbonyl bands change around 3400 cm^{-1} and 1700 cm^{-1}, confirming the changes in the molecular structure due to doping. The features of these bands (metal–hydroxyl formations) suggest the increased functionality of the surface in the MAC sample. Such surface modifications are known to improve catalytic activity in transesterification reactions.The inclusion of iron oxide in the activated carbon structure provides several benefits for catalysis. Due to their magnetic property, the catalyst can be easily separated by an external magnetic field after reaction, reducing cost and improving reusability. The kinetics of the reaction are also promoted by triglyceride and alcohol adsorption over iron-based compounds [13].

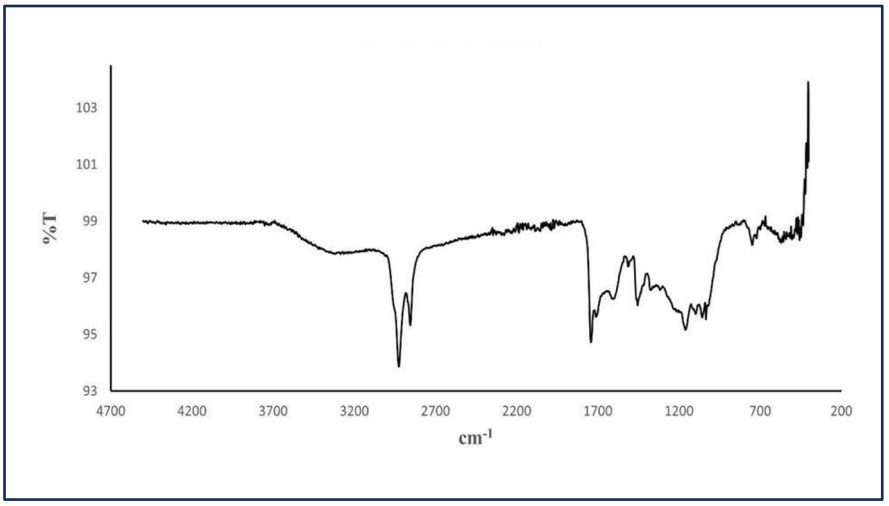

Fig. 2. FTIR analysis of activated carbon

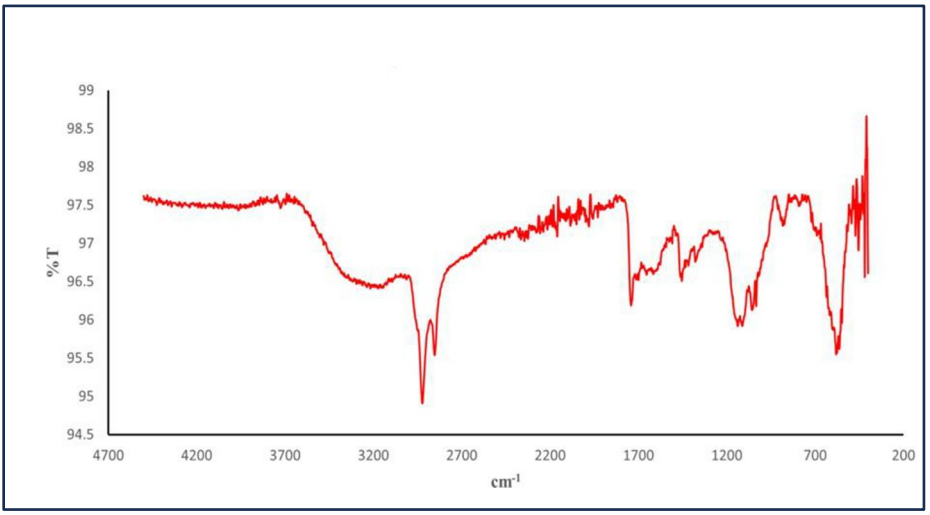

Fig. 3. FTIR analysis of magnetised biocatalyst

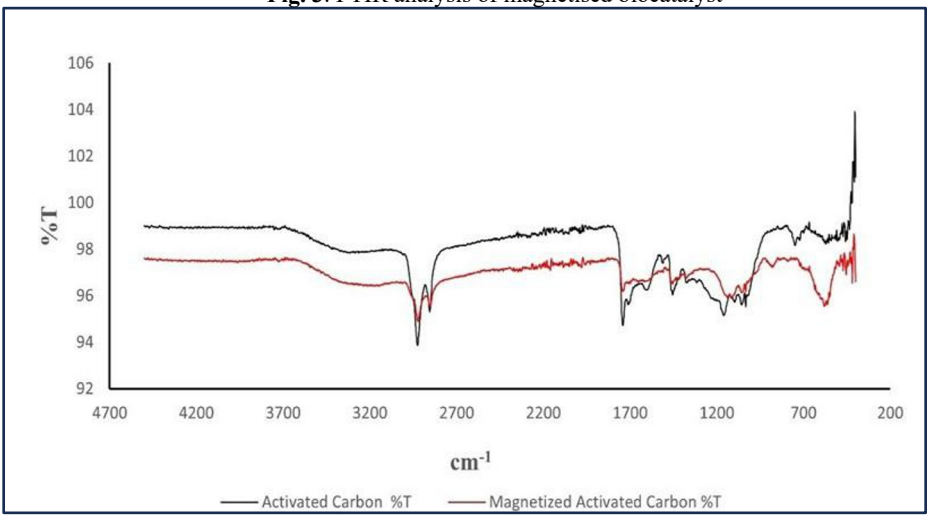

Fig. 4. FTIR analysis comparison on doping

3.2 X–Ray Diffraction

X-ray Diffraction (XRD) characterisation was performed to analyse the crystal structure of the activated carbon (AC) and magnetised activated carbon (MAC). The AC diffraction pattern (Fig. 5) shows a broad peak centred at $2\theta \sim 23°–25°$, associated with the (002) plane of amorphous graphitic carbon [14]. This supports the amorphous structure of pomegranate seed waste–based activated carbon. The lack of sharp peaks also indicates the poor ordering and the porous nature of the material [14].

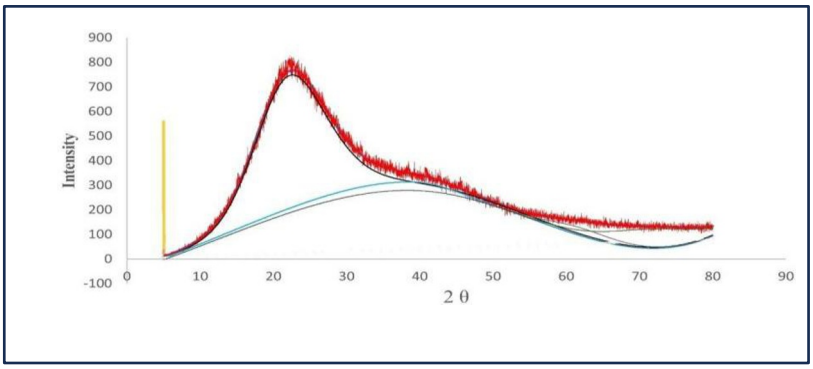

Fig. 5. XRD analysis of activated carbon

The XRD pattern of the MAC sample (Fig. 6) shows extra sharp peaks at about 2θ = 30.1° and several other characteristic lines corresponding to magnetite (Fe_3O_4) [15]. The presence of these crystalline peaks shows that the incorporated iron oxide nanoparticles were successfully formed on carbon. The carbon hump is still visible, which indicates that the magnetic functionalisation has not substantially modified the amorphous carbon matrix. The lack of hematite or maghemite peaks also implies that the magnetite formed is of high purity [15]. The combination of the crystalline magnetite peaks and the amorphous carbon background indicates a dual-phase structure made up of a high-surface-area amorphous carbon matrix with immobilised magnetic sites [16]. During heterogeneous processes, this structure facilitates both the material's magnetic recovery and catalytic activity.

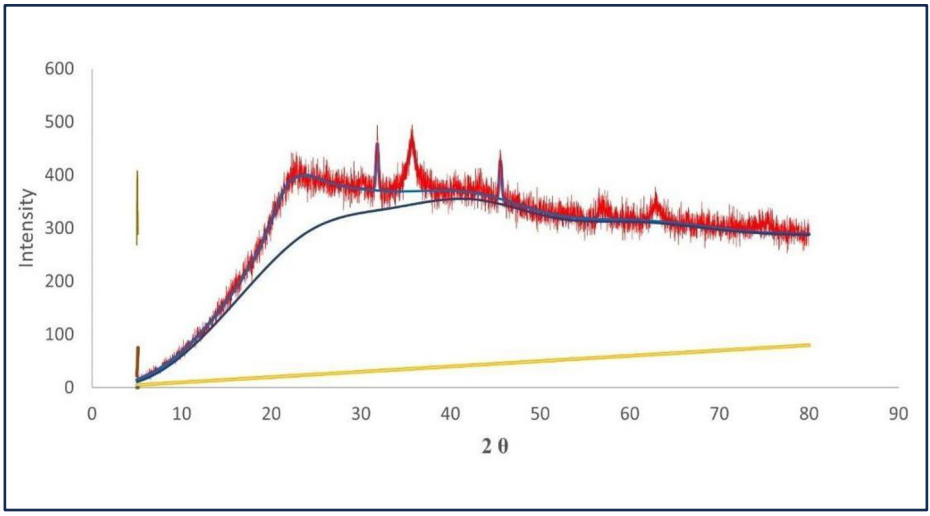

Fig. 6. XRD analysis of magnetised activated carbon

4 Conclusion

The current study has successfully prepared a heterogeneously magnetised activated carbon–supported catalyst derived from pomegranate seed biomass. The general goal was to produce a magnetically active, solid and green catalyst, while being cost-effective, which could bring about an improvement in some of the drawbacks in homogeneous catalyst, such as separation and recovery, as well as reusability and sensitivity to pollutants. The project aimed at the development of an agricultural residue-to-high–surface-area porous catalyst support taking advantage of biomass valorisation. A catalyst having the properties of being a dual functional material was achieved when the carbon structure was activated and magnetically functionalized, for the purpose of applying it in heterogeneous catalysis. The carbon support not only acted as a thermally stable and chemically inert host matrix, but also facilitated the excellent dispersion of active magnetic sites, which could give rise to the increased catalytic surface exposure and magnetic recoverability. The successful preparation of the catalyst was verified by contemporary characterisation techniques. FTIR study confirmed the existence of important surface functional groups, and the XRD data confirmed the formation of crystalline magnetite in the carbon matrix, which revealed a great extent of integration with magnetic particles. These characterisation studies confirmed the design of the catalyst and gave us an understanding of its physicochemical features with respect to catalysis. According to these results, the material has a potential applicability in transesterification and other green catalytic processes.

A key advantage of the developed catalyst is its magnetic property, which enables rapid and efficient recovery using an external magnet, offering significant operational benefits for scalability. From an environmental and economic standpoint, the use of biowaste as a precursor and the avoidance of hazardous reagents align well with green chemistry and circular economy principles.

This catalyst demonstrates potential for broader applications, including glycerol upgrading, methanolysis-based hydrogen generation, and treatment of industrial or pharmaceutical wastewater, where magnetically separable catalysts are highly desirable. Future techno-economic assessments may support the applicability of this material in decentralised or distributed catalytic systems.

In summary, the magnetised activated carbon synthesised in this study exhibits magnetic recoverability, large surface area, chemical stability, and an eco-friendly synthesis route, making it a promising material for future use in sustainable energy and environmental applications. These findings provide a strong foundation for pilot-scale development and contribute to the ongoing shift toward low-carbon and resource-efficient technologies.

Disclosure of Interest. The authors have no competing interests to declare that are relevant to the content of this article.

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