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Submission date: 29-Sep-2025 09:18PM (UTC-0500)
Submission ID: 2766342784
File name: STUDY_OF_FE_O_-COCONUT_SHELL_CHARCOAL_AS_A_CARBON.docx (1.06M)
Word count: 2741
Character count: 16005

PREPARATION STUDY OF Fe_3O_4-COCONUT SHELL CHARCOAL AS A CARBON QUANTUM DOTS CQDS BASED NANOPARTICLE MATERIAL

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Article History
Received: XX XXXXXXX XXX; Received in Revision: XX XXXXXXX XXX; Accepted: XX XXXXXXX X

Abstract (Verdana 8 font)

This research aims to study the preparation of Fe_3O_4 nanoparticles based on coconut shell charcoal as a carbon source for Carbon Quantum Dots (CQDs) using the hydrothermal method. The synthesis of Fe_3O_4 nanoparticles was carried out through the coprecipitation method by mixing $FeCl_3 \cdot 6H_2O$ and $FeSO_4 \cdot 7H_2O$ solutions while stirring at 60°C, followed by the addition of CQD solution derived from coconut shell charcoal. The variable parameters include the mass of coconut shell charcoal, reaction temperature, and precursor concentration. Characterization was performed using X-ray Diffraction (XRD) to determine the optical properties, crystal structure, and magnetic properties of the resulting nanoparticles. XRD analysis confirmed that the synthesized Fe_3O_4-CQD nanoparticles exhibit a cubic spinel crystal structure and superparamagnetic properties, making them highly suitable for applications in bioimaging and catalysis. This research contributes to the utilization of organic waste as a raw material for environmentally friendly nanomaterials and has the potential to enhance the economic value of coconut waste.

Keywords: *Fe_3O_4, CQDs, crystal structure, nanopartikel, hydrotermal,*

Abstrak

Penelitian ini bertujuan untuk mempelajari preparasi nanopartikel Fe_3O_4 berbasis arang tempurung kelapa sebagai bahan sumber Carbon Quantum Dots (CQDs) melalui metode hidrotermal. Sintesis nanopartikel Fe_3O_4 dilakukan melalui metode kopresipitasi dengan mencampurkan larutan $FeCl_3 \cdot 6H_2O$ dan $FeSO_4 \cdot 7H_2O$ sambil diaduk pada suhu 60°C, kemudian ditambahkan larutan CQDs yang diperoleh dari arang tempurung kelapa. Parameter variabel meliputi massa arang kelapa dan suhu reaksi, serta konsentrasi bahan prekursor. Karakterisasi dilakukan menggunakan X-ray Diffraction (XRD) untuk mengetahui sifat optik, struktur kristal, dan sifat magnetik nanopartikel yang dihasilkan. Analisis XRD menegaskan bahwa nanopartikel Fe_3O_4-CQD yang disintesis memiliki struktur kristal spinel kubik dan sifat superparamagnetik, menjadikannya sangat cocok untuk aplikasi dalam bioimaging dan katalisis. Penelitian ini memberikan kontribusi dalam pemanfaatan limbah organik sebagai bahan dasar pembuatan nanomaterial yang ramah lingkungan dan berpotensi meningkatkan nilai ekonomi limbah kelapa.

Keywords: *Fe_3O_4, CQDs, struktur kristal, nanopartikel, hidrotermal*

1. Introduction

The escalating issue of water pollution poses a significant challenge to environmental sustainability, particularly in regions with intensive industrial and agricultural activities. Such waste contains various pollutants that can degrade ecosystems and threaten human health if not properly managed. Conversely, solid waste like coconut shells, which are abundant in tropical areas such as Banyuwangi, is often discarded without optimal utilization. In fact, coconut shells possess considerable potential as a raw material for activated carbon production due to their superior physical and chemical properties, including high hardness, large surface area, excellent adsorption capacity, and low ash content with high purity (Masriatini and Fatimura, 2019). By utilizing coconut shells as a source of activated carbon, solid waste can

be transformed into high-value products while simultaneously contributing to effective mitigation of water pollution.

Activated carbon derived from coconut shells exhibits pore characteristics and surface area that critically influence its adsorption capacity (Latifah, 2020). The smaller the particle size of the activated carbon, the greater its surface area, thereby enhancing its ability to adsorb pollutants (Saputro et al., 2019). The development of nanoparticle technology has emerged as a key strategy to improve the efficiency of activated carbon. Nanoparticles, typically ranging from 1 to 100 nanometers in size, can enhance the bioavailability and stability of active substances, as well as optimize the delivery systems for targeted applications (Abdassah, 2017). Surface modification of nanoparticles with organic or inorganic materials such as ZnO can further improve the optical properties and stability of Fe_3O_4 magnetic nanoparticles, thereby enhancing their performance in catalytic and bioimaging applications (Winataputra et al., 2014; Khaira et al., 2022; Veronica et al., 2022).

One of the latest innovations involves the synthesis of Carbon Quantum Dots (CQDs) from coconut shell charcoal using the hydrothermal method, yielding carbon nanoparticles smaller than 10 nm with excellent photoluminescent properties (Wahyuni, Afthoni, and Rollando, 2022).

These CQDs exhibit notable advantages such as biocompatibility, photostability, and water solubility, making them promising materials for sensor and optical contrast applications (Triwardiati and Ermawati, 2018). Other studies have also demonstrated that CQDs derived from natural sources such as lemon and grape extracts possess an absorption spectrum around 350 nm, corresponding to n-n* electronic transitions (Fini et al., 2018). By integrating the potential of coconut shell waste as a source of activated carbon with advanced nanoparticle technology, innovative solutions for waste management and the development of environmentally friendly functional materials can be realized—particularly in resource-rich regions like Banyuwangi.

2. Methodology

2.1 tools and material

60 mesh sieve, Memmert oven, pyrolysis instrument, hot plate, magnetic stirrer, measuring pipette (pyrex), glass funnel (pyrex), beaker glass (pyrex), measuring cup (pyrex), analytical balance, (Pioneer), watch cup, and XRD instrument (PANalytical).

2.2 material

Coconut Shell Activated Charcoal 50 gr, 100 mL Phosphoric Acid (H_3PO_4) 85% and Sodium Chloride (pa) 4 gr, Aquadest, Filter paper, iron (III) chloride hexahydrate ($FeCl_3$-$6H_2O$), iron (II) sulfate heptahydrate ($FeSO_4.7H_2O$), ammonium hydroxide (NH_4OH) 30 mL.

2.3 Research method

2.3.1 Synthesis of Activated Charcoal CQDs

50 grams of 60 mesh coconut shell charcoal were soaked each in 100 mL of 4% H_3PO_4 and 4% NaCl solution for 24 hours. The mixture was filtered and washed with distilled water until clean, placed in the oven at a temperature of around 110°C for 2 hours. After drying, the charcoal is cooled in a desiccator. In the next process, the charcoal is pyrolyzed for 2.5 hours at a temperature of 400°C. 0.3 grams of carbon powder resulting from pyrolysis was then dissolved in 20 mL of distilled water, then the solution was filtered using filter paper to obtain colloidal CQDs.

2.3.2 Synthesis of Fe_3O_4 Nanoparticles

In this method, 8.109 grams of $FeCl_3 \cdot 6H_2O$ and 4.170 grams of $FeSO_4 \cdot 7H_2O$ are dissolved in 30 mL of distilled water, then the solution is heated on a hot plate until it reaches a temperature of 60°C. After that, 30 mL of NH_4OH solution was added little by little to the solution while stirring using a magnetic stirrer for 90 minutes (Novita et al, 2023).

Commented [r1]: Adds the chemistry reaction mechanism

Chemistry reaction mechanism:

Hydrolysis of NH_4OH: NH_4OH provides the necessary OH^- ions in the solution.

$$NH_4OH \rightleftharpoons NH_4^+ + OH^-$$

Nucleation and Precipitation: The hydroxide ions (OH^-) react with the Fe^{3+} and Fe^{2+} ions to form an intermediate mixed precipitate of ferric hydroxide ($Fe(OH)_3$) and ferrous hydroxide ($Fe(OH)_2$).

$$Fe^{3+} + 3OH^- \rightarrow Fe(OH)_3$$
$$Fe^{2+} + 2OH^- \rightarrow Fe(OH)_2$$

Formation of Magnetite (Fe_3O_4): Under the influence of heat and the aqueous environment, the intermediates undergo an oxidation-reduction and dehydration process to form the final magnetite nanoparticles. The overall reaction can be summarized as:

$$Fe(OH)_2 + 2Fe(OH)_3 \rightarrow Fe_3O_4 + 4H_2O$$

The prolonged stirring ensures a homogeneous mixture and controls the growth of the nanoparticles, leading to a more uniform particle size distribution. The resulting black precipitate is the magnetic Fe_3O_4 nanoparticles.

2.3.2 **Synthesis of Fe_3O4-CQDs Nanocomposites**

Fe_3O_4-CQDs nanocomposites were synthesized via a hydrothermal method, where the solution mixture was placed in an oven and heated at 80°C for 12 hours. The crystal size of the Fe_3O_4-CQDs nanocomposite samples was determined using the Debye Scherrer equation, which refers to peak broadening in the X-ray diffraction pattern. Mathematically, the Debye Scherrer Equation can be formulated as Equation 1.

$$B = \frac{K\lambda}{B\ cos\theta} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

D : Crystal dimensions in nanometers (nm)
λ : Wavelength of X-ray radiation used
K: Crystal form factor (usually has a value between 0.9 to 1)
B : Width of the diffraction peak at half maximum intensity, usually called FWHM (Full Width at Half Maximum), in units of degrees
cos θ : Cosine of the angle between the incident direction of the X-rays and the normal direction of the crystal plane

2.4 Research Design

The research, "Preparation Study of Fe_3O_4-Coconut Shell Charcoal as a Carbon Quantum Dots CQDS-Based Nanoparticle Material," is an experimental research study. Fe_3O_4 nanoparticles were prepared using a coprecipitation method, which involved mixing $FeCl_3 \cdot 6H_2O$ and $FeSO_4 \cdot 7H_2O$ chemicals, then adding a base solution of NH_4OH to trigger particle formation. After formation, the surface of the magnetic nanoparticles was modified using carbon quantum dots (CQDs), a luminescent material, to enhance their function. characterization process using characterization XRD

3. Results and Discussion

3.1 Synthesis of Activated Charcoal CQDs

Based on figure 1. it shows that the sample was cooled, then pyrolyzed for 2.5 hours at a temperature of 500 oC. After obtaining the pyrolysis sample, the researcher weighed 0.3 g of carbon powder and dissolved it in 20 ml of distilled water. The solution was filtered using filter paper to produce colloidal CQDs.

Figure.1 CQDs colloids

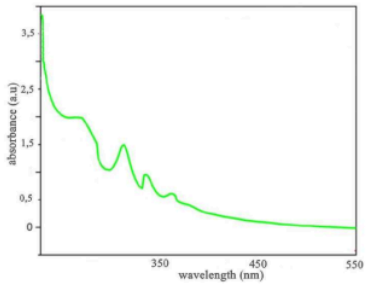
3.2 Sintesis Nanopartikel Fe_3O_4–CQDs

According to Wahfiudin, in 2025, the sample mixing reaction will show the chemical reaction of the Fe_3O_4-CQDs nanocomposite. The interaction between the functional groups of CQDs and Fe_3O_4. The interaction in question refers to the relationship or reaction between the functional groups on the surface of CQDs (-COOH and -OH) and the surface of Fe_3O_4. This interaction is important because it determines the physical and chemical properties of the resulting nanocomposites such as stability, photocatalytic activity and magnetic separation ability.

Figure 2. a. FE_3O_4 b. Fe_3O_4-CQDs

CQDs contain carboxyl (-COOH) and hydroxyl (-OH) functional groups on their surface which are formed from a synthesis process using natural materials. On Fe_3O_4 nanoparticles, the iron oxide surface (≡Fe-O-) will form coordination bonds with these groups, in this case the oxygen atom from the carboxyl or hydroxyl group on CQDs can act as an electron pair donor, while the Fe ion in Fe3O4 acts as an acceptor to form a strong coordination bond, through a hydrogen bonding mechanism between the hydroxyl group (-OH) of Fe_3O_4 which is strongly bound to the carboxyl group (-COOH) and hydroxyl groups (-OH) belong to CQDs. These hydrogen bonds strengthen the bond between the two materials and influence the stability and surface properties of the nanocomposite. And through the electrostatic interaction mechanism, there is an attractive force between the surface charges of the two materials which plays an important role in the formation and stability of nanocomposites, especially if the surfaces of the two materials have opposite charges. This process produces magnetic-luminescent nanocomposites (Fe_3O_4-CQDs) with potential applications in photocatalysis and environmental remediation, exploiting the photocatalytic properties of CQDs and the ease of magnetic separation of Fe_3O_4.

4.4 FTIR Spectrum Analysis
Tingginya nilai absorbansi CQD mencerminkan konsentrasi CQD yang terbentuk dalam jumlah signifikan.



4.4 X-Ray Diffraction (XRD) Instrument Analysis

Based on the XRD data analysis results from the CQDs-Fe3O4 sample, the data obtained were in the form of a spectrum graph as shown in Figure 4.1.

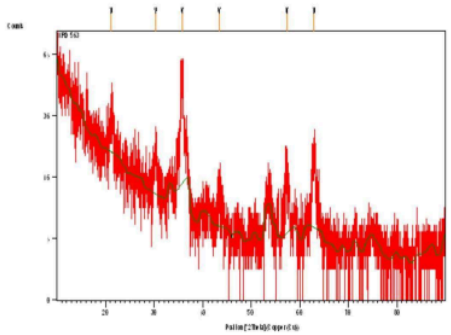


Figure .3 X-ray diffraction pattern of CQDs-Fe3O4 sample

Table. 1 Results of XRD analysis of the sample and estimated crystallite size produced.

No.	top peak (°2θ)	FWHM (°2θ)	θ (°)	θ (rad)	FWHM (rad)	cos θ	Crystal size (mm)
1	211,93	0,63	105,95	0,18	0,01	0,98	12,8
2	302,98	0,79	151,49	0,26	0,01	0,96	10,3
3	356,94	0,47	178,46	0,32	0,01	0,95	17,7
4	432,78	0,63	216,39	0,37	0,01	0,93	13,5
5	573,71	0,62	286,85	0,51	0,01	0,88	14,3
6	629,57	0,96	314,78	0,55	0,02	0,85	8,7

The results of X-ray Diffraction (XRD) analysis of Fe_3O_4-CQDs nanocomposites show six main peaks that reflect the structure and characteristics of the material formed. The peak at 21.19° 2θ (d=4.19263 Å) attracts attention because it does not match the standard pattern of pure Fe_3O_4 (JCPDS 19-0629) and has a significant intensity (32.01%). This is thought to originate from the interaction between carbon quantum dots (CQDs) with the Fe_3O_4 surface, or the possible presence of a minor phase of goethite (α-FeOOH) which has a similar angle. Meanwhile, five other peaks, namely at 30.30°; 35.69°; 43.28°; 57.37°; and 62.96° 2θ, which correspond well to the crystallographic planes of Fe_3O_4, representing the (220), (311), (400), (511), and (440) planes, respectively. The main peak at 35.69° 2θ (d=2.51552 Å) has the highest intensity (100%) and the narrowest FWHM (0.4723°), indicating a high degree of crystallinity with an estimated crystallite size of around 25-30 nm. Other peaks also show nanoparticle sizes (<50 nm) with varying FWHM, indicating a non-uniform particle size distribution.

In general, this XRD pattern confirms the dominance of the magnetite phase (Fe_3O_4) in the nanocomposite structure, where five of the six main peaks can be identified as characteristic of Fe_3O_4. For more details, we show the graph and table of the XRD peaks of Fe_3O_4.

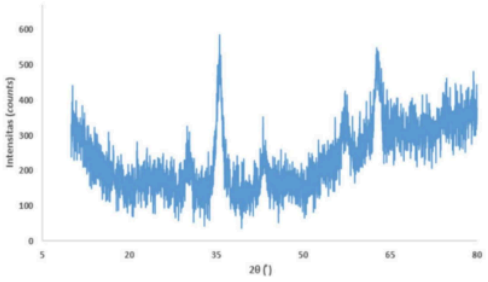


Figure 4. XRD crystal structure of magnetite Fe_3O_4

In general, this XRD pattern confirms the dominance of the magnetite phase (Fe_3O_4) in the nanocomposite structure, where five of the six main peaks can be identified as characteristic of Fe_3O_4. For more details, we show the graph and table of the XRD peaks of Fe_3O_4.

Table. 2 XRD spectrum peak identification table

No.	2 θ°	Miller Index (hkl)	Priority
1	30.01	-220	Top peak magnetit
2	35.6	-311	Highest intensity peak
3	43.09	-400	
4	53.78	-422	
5	57.24	-511	
6	62.68	-440	
7	74.73	-533	
8	79.35	-622	

These peaks indicate the spinel cubic (Face Centered Cubic, FCC) crystal structure of magnetite Fe_3O_4. The influence of CQDs is clearly visible through the shift of the 2θ angle and the decrease of the peak intensity, which indicates a modification in the lattice parameters due to the surface interaction between Fe_3O_4 and CQDs. The wide FWHM of several high-angle peaks also indicates that the resulting material is nano-sized with a fairly varied size distribution.

4. Conclusion

The characterization results show that CQDs produced from coconut shell charcoal have an optimal absorbance spectrum around 350 nm and an amorphous structure according to XRD analysis. Meanwhile, Fe_3O_4 nanoparticles were successfully synthesized with a particle size of around 13 nm, which is very suitable for biomaging and catalysis applications.

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