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Study of Magnetic Nanoparticles (Fe_2O_3 and Fe_3O_4) Synthesized by Electric ARC - Discharge Technique

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Abstract

Magnetic nanoparticles based on Fe2O3 (Hematite) and Fe3O4 (Magnetite) are synthesized with an average diameter ranging from 30 nm to 45 nm using the Electric Arc Discharge Technique at a voltage of 70 V. These nanoparticles exhibit diverse applications in fields such as ferrofluids, magnetic refrigeration, catalysis, biomedicine, chemotherapy, magnetic hyperthermia, infertility treatments, cell isolation, medical diagnosis, magnetic gels, memory cards, memory chips, and various other biomedical applications. Worldwide, there is significant effort being directed toward developing simple, cost-effective methods for mass production of magnetic nanoparticles using electrolytes based on LiCl, KCl, and NaCl salts. This research focuses on the production of these nanoparticles, with the samples being characterized using techniques such as Vibrating Sample Magnetometry, X-Ray Diffraction, and Scanning Electron Microscopy.

Keywords

Hematite, Magnetite, Nanoparticles, Electrolytes, Characterization.

INTRODUCTION

Nanotechnology is an emerging field that has captured the attention of researchers worldwide. It focuses on materials at the nanoscale, which exhibit unique properties not seen in their bulk 3-dimensional counterparts. These exceptional characteristics arise from (1) the large surface area, (2) the modification of electronic structures, and (3) the symmetry-breaking associated with low-dimensional materials. These fundamental changes contribute to enhanced mechanical, electrical, magnetic, chemical, thermal, and optical properties. As a result, nanotechnology has seen rapid growth, with its impact evident in the reduction of size and weight of devices, as well as significant improvements in accuracy, consistency, and data transmission rates. These advantages, which are not observed in traditional bulk 3D materials, have propelled advancements in the field. Consequently, various materials such as metals, alloys, oxides, carbides, nitrides, polymers, biomaterials, and nanocomposites are being studied in their low-dimensional forms. They are synthesized using a range of techniques and applied in diverse fields, including medicine, water purification, sensors, space exploration, data storage, information technology, and cosmetics, among others.

In the pursuit of exploiting the benefits of easily accessible, cost-effective, and abundant iron oxides like magnetite (Fe3O4) and hematite (Fe2O3), these materials have been extensively studied in their nanoscale form due to their magnetic properties[1], including superparamagnetic. Their wide array of applications includes magnetic liquids[2] for energy, magnetic refrigeration[3-4], catalysis[5], biomedicine[6], chemotherapy[7], infertility treatments[8], cell isolation[9], magnetic nanofluid hyperthermia[10], and medical diagnosis[11]. To meet the demands of such diverse applications, these magnetic nanoparticles have been synthesized using various techniques, which are generally classified into top-down and bottom-up approaches.

EXPERIMENTAL PROCEDURE

Magnetic nanoparticles are synthesized using the electric arc - discharge technique with 2 mm (Diameter) iron rod electrode of ER-70S2. An individual aqueous solution with one mole of each NaCl (58.44 gm/mole), KCl (74.55 gm/mole), or LiCl (42.39 gm/mole) is made with 1000 ml of distilled water as an electrolyte in a 2000 ml capacity glass beaker where in the electrodes are spaced 3 cm apart and dipped 6 cm in the electrolyte. To avoid any explosion while conducting the experiment, the beaker is encompassed by ice. The essential features of the set-up used are shown in Figures 1 and 2.

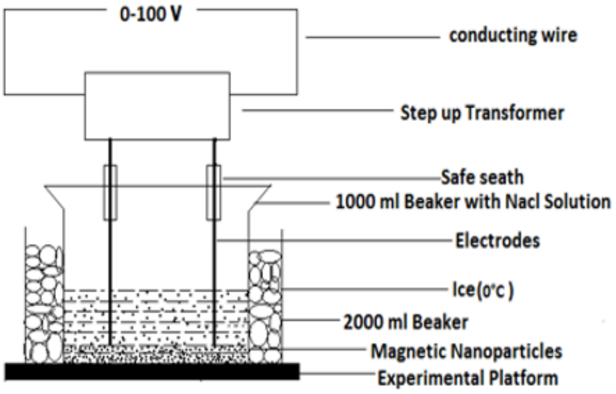

Figure 1. Experimental Set-up (Line Diagram)

Figure. 2. Experimental Set-up

As the current is supplied to the electrodes at 70 V, the anode starts dissolving in the electrolyte. It has been observed that the electrolysis process slows down after 10-12 minutes, which indicates that the supply of current must be regulated. Now the nanopowder procured is isolated from the electrolyte by using a sieve paper and the obtained powder is cleansed thoroughly with H_2O and spirit (absolute alcohol) to eradicate the existence of salts. To convert the amorphous state of the material to crystalline, heat treatment is to be done at 75°C for 10 hours in a muffle-cum-tube furnace. Later, the powder is stored in air-tight receptacles to prevent any further reactions with ambient air [12].

RESULTS AND DISCUSSION

I. X-Ray Diffraction:

Structural properties of the materials were investigated using XRD measurements technique. Fig's. 3(a, b), 4(a, b), 5(a, b) depicts the comparative XRD patterns of KCl, NaCl, and LiCl electrolytes with magnetic nanoparticles at 70 V before and after heating [13]. The above-following mentioned images depict XRD patterns obtained with an ER70S-2 electrode in various electrolytes. In this work, we used KCL, NaCl, and LiCl as electrolytes for comparative study.

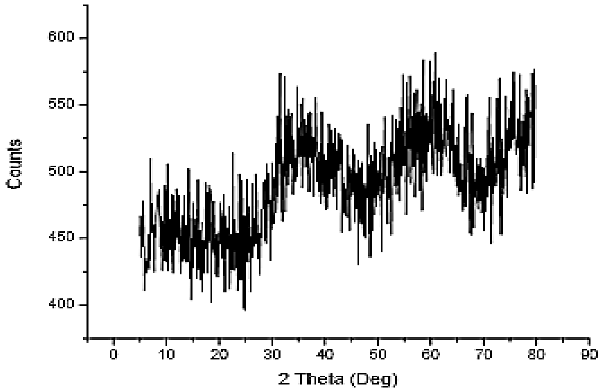

Figure. 3-a. NaCl electrolyte's XRD before heat treatment

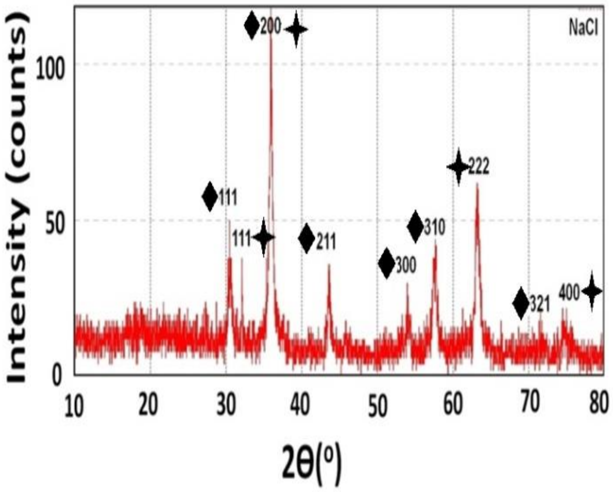

Figure. 3-b. NaCl electrolyte's XRD after heat treatment

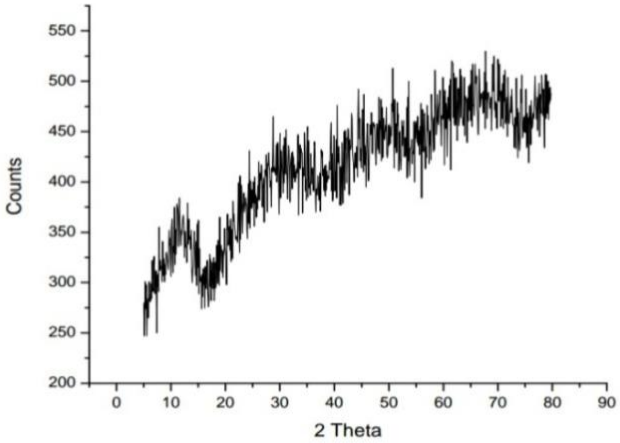

Figure. 4-a. KCl electrolyte's XRD before heat treatment

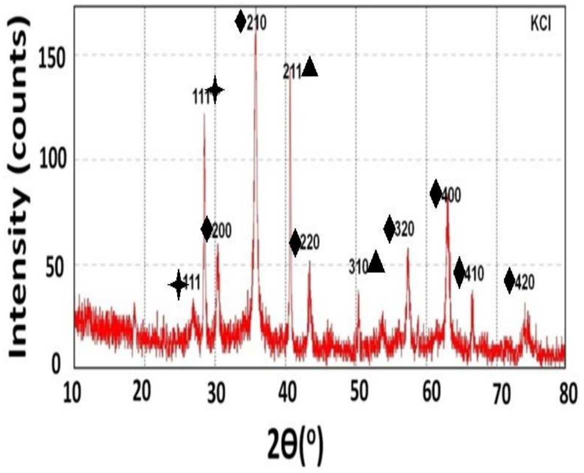

Figure. 4-b. KCl electrolyte's XRD after heat treatment

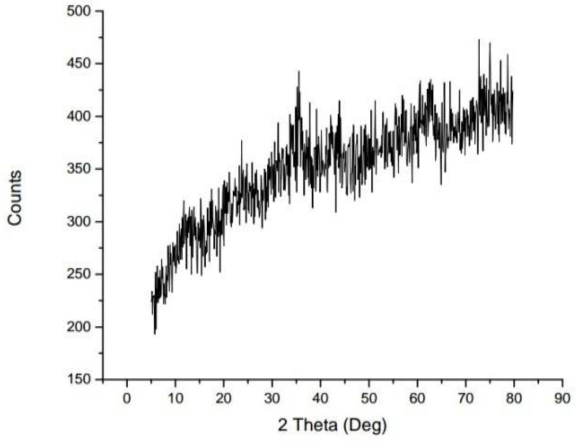

Figure. 5-a. LiCl electrolyte's XRD before heat treatment

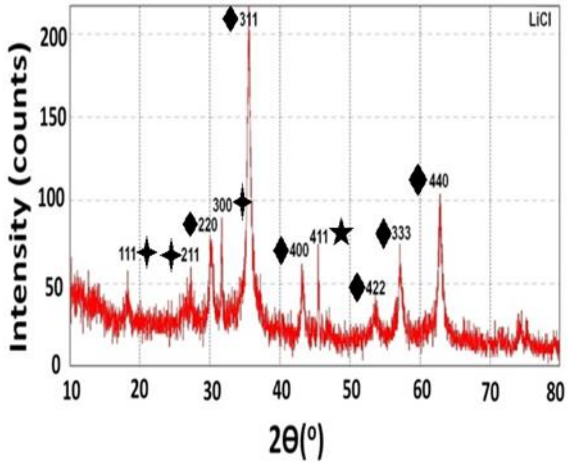

Figure. 5-b. LiCl electrolyte's XRD after heat treatment

It was observed that there were no powerfully built peaks in the figures (3-a, 4-a & 5-a) XRD results, specifying the presence of amorphous structure. In this study, the XRD patterns in different electrolytes after processing with the heat treatment were shown in figures (3-b, 4-b & 5-b) with many strong peaks which indicate the structure to be crystalline. For NaCl, KCl and LiCl, peak values were found to be 200, 210 and 311 respectively.

The XRD technique also detects iron oxide and iron residues. The patterns in Figures 3-b, 4-b, 5-b, show many strong peaks which indicate that the structure obtained to be crystalline. As per the obtained data of the 2θ values, the phases are illustrated in Table-1. The peaks were recognized as Fe_2O_3 and Fe_3O_4. 2θ = 35.53^0 is a quality 2θ for both Fe_2O_3 and Fe_3O_4 nanoparticles. Fe & FeO nanoparticle traces were also found in the X-ray diffraction.

Table 1: Magnetic nanoparticles with standard 2θ angle.

NAME	2θ	PHASE NAME	2θ	NAME	2θ
Fe_2O_3 ✦	17.90 27.20 33.20	Fe3O4 ◆	30.16	Fe ★	
			35.53		**45.30**
			43.18		
	35.53		53.40		
	38.93		57.11		
	45.20		62.71	▲ FeO	
	54.20		66.20		**41.25**
	63.80		71.00		**50.81**
	75.30		74.40		

Particle size calculation:

The average nanoparticle size is evaluated by the following Debye-Scherrer equation [13] and data is mentioned in Table -2.

$$D = \frac{0.9\lambda}{d\cos\theta} \quad \text{------------------(1)}$$

Here, λ= Wavelength (0.154 nm).

θ = Bragg's Diffraction angle.

d= FWHM (full width at half maximum intensity of the peak in radians).

D = Particle size in nm.

Table 2: Magnetic nanoparticles size evaluated using equation 1.

Salts	Minimum (nm)	Maximum (nm)	Average (nm)
NaCl	18.34	30.03	24.32
KCl	20.95	36.42	28.68
LiCl	15.46	29.71	22.58

From table 2, the mean size of the nanoparticles found was less than 40 nm using equation (1). It is also noted that the nanoparticles synthesized using LiCl electrolyte have the size as low as 15.46 nm. Table-2 reveals the fact of the benefit of LiCl salt electrolyte to produce fine smaller nanoparticles when compared with the other electrolytes.

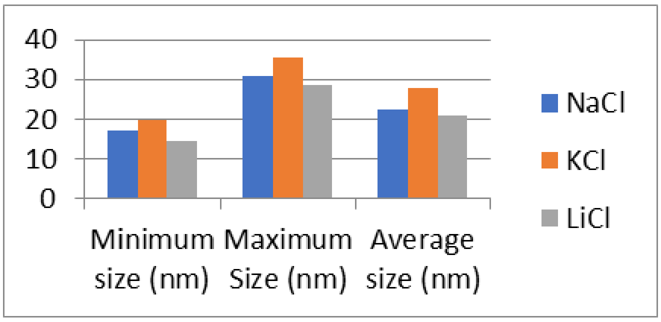

Figure. 6. Graphical representation of particle size calculated by Debye-scherrer formula.

II. Scanning Electron Microscope:

Microscopy is a crucial characterization technique for determining the properties of nanoparticles [14]. The resolving power of a light microscope is limited due to the numerical aperture and illumination wavelength of the lens. In contrast, electron microscopes offer significantly higher resolving power by using an electron beam instead of light. Scanning Electron Microscopes (SEMs) are a type of high-resolution electron microscope that employ a focused electron beam to produce images of the sample with a resolution down to a few nanometers.

In the current study, a scanning electron microscope (S-3700N with an accelerating voltage of 30 kV) is utilized to determine the size of magnetic nanoparticles. Table 3. shows the lowest and maximum size of the nanoparticles at 70 V. Figures 7 – 9, show the SEM image findings, which include particle sizes[14]. These findings back up the particle sizes predicted by XRD.

SEM report of magnetic nanoparticles of ER70S-2 with different electrolytes at an operating voltage of 70 V.

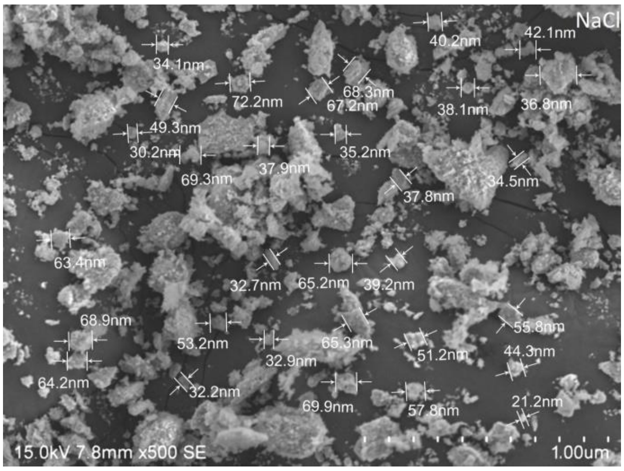

Figure.7. SEM report of magnetic nanoparticles found in NaCl electrolyte.

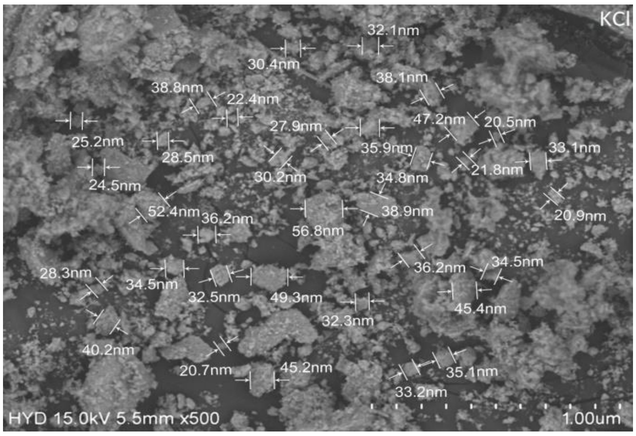

Figure. 8. SEM report of magnetic nanoparticles found in KCl electrolyte.

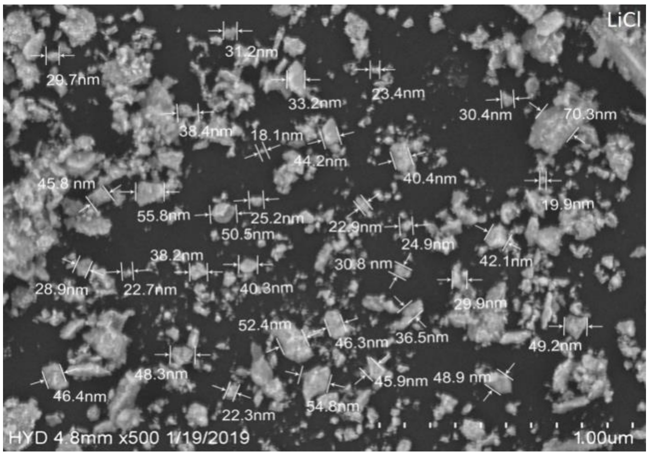

Figure. 9. SEM report of magnetic nanoparticles found in LiCl electrolyte.

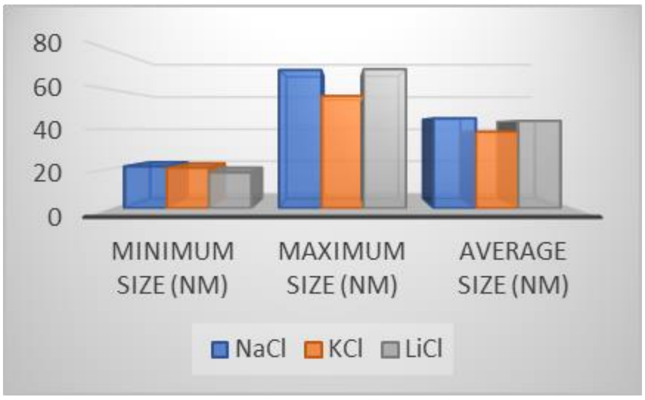

Figure. 10. Graphical representation of magnetic nanoparticle size through SEM

Table 3: Maximum, Average and Minimum size of magnetic nanoparticles using Scanning Electron Microscope.

Salts	Minimum (nm)	Maximum (nm)	Average (nm)
NaCl	20.25	70.98	45.61
KCl	21.56	58.89	40.22
LiCl	19.17	68.31	43.74

III. Vibrating Sample Magnetometer results of ER70S-2 based magnetic nanoparticles using different electrolytes at an operating voltage of 70 V.

This instrument is extensively used for the magnetic characterization of a broad range of materials and is known as a vibrating sample magnetometer (VSM). In this way, the magnetic characterization of materials may be determined as a function of the applied magnetic field, time, and temperature.

The graph between applied field and magnetic moment may be used to determine the magnetic characteristics of materials using a vibrating sample magnetometer (G). The graph identifies the parameters saturation magnetization (M_s), magnetic remanence (M_r), squareness ratio (SQR) and coercivity (H_c). Magnetic nanoparticles are employed in memory storage devices because their application is based on the value of SQR, i.e., if the SQR is under 0.5, it implies tiny single domain [15].

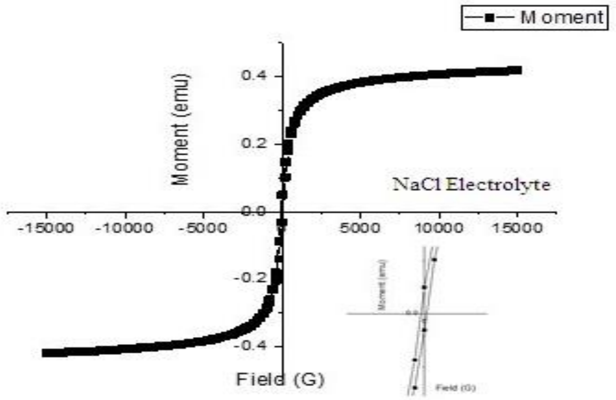

Figure. 11. NaCl Electrolyte based Hysteresis Curve of ER70S-2 based magnetic nanoparticles.

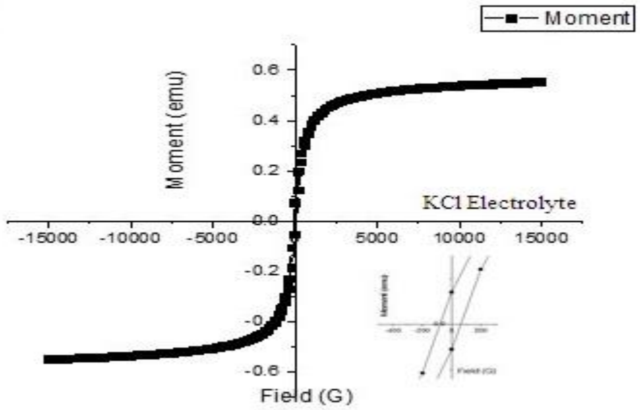

Figure. 12. KCl Electrolyte based Hysteresis Curve of ER70S-2 based magnetic nanoparticles.

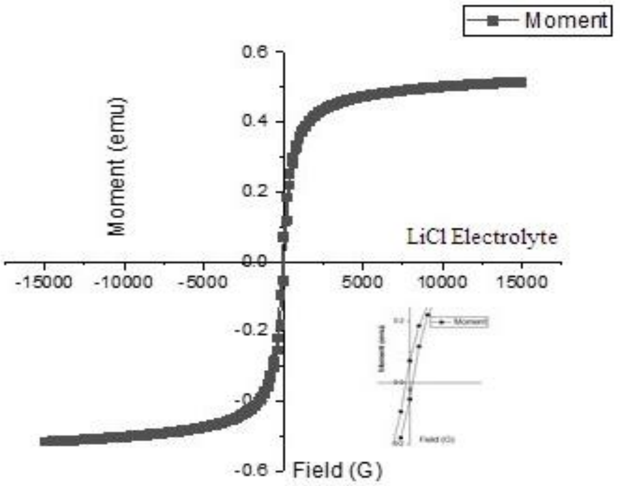

Figure. 13. LiCl Electrolyte based Hysteresis Curve of ER70S-2 based magnetic nanoparticles.

Figures 11 - 13, depict a graphical depiction of the hysteresis curve of magnetic nanoparticles for various electrolytes and temperatures. Magnetic characteristics of magnetic nanoparticles utilizing different electrolytes are shown in Table-4.6. With reference to Table 4, it is evident that the highest value of magnetic remanence (M_r) is 0.708 (KCl) and the lowest value is 0.0494 (NaCl). The maximum saturation magnetization (M_s) value is 0.5530 (KCl), while the minimum saturation magnetization (M_s) value is 0.4193 (NaCl). When using KCl, the coercivity (H_c) value is 62.053. When using NaCl, the coercivity (H_c) value is 45.504. Finally, the highest value for squareness ratio (SQR) is 0.132 for LiCl and the lowest value is 0.11 for NaCl[15].

Table 4: Magnetic nanoparticles properties using different Salts.

Parameters	**NaCl**	**KCl**	**LiCl**
Remanence	0.049	0.070	0.070
Saturation Magnetization	0.419	0.553	0.514
Coercivity	45.50	62.05	61.54
Squareness ratio	0.11	0.124	0.132

Comparison of electrolyte in terms of quantity and production cost.

Our preliminary research has demonstrated the benefits of using Potassium Chloride (KCl) electrolytes for producing magnetic nanoparticles, particularly in terms of cost-effectiveness and faster production compared to other electrolytes such as NaCl and LiCl. Producing 100 grams of magnetic nanoparticles takes approximately 90 minutes and costs around $10, while the same quantity is sold commercially at 20 to 25 times the production cost. This analysis clearly shows that the Electric Arc Discharge technique offers an economical and commercially viable method for nanoparticle production.

Table 5: Comparison of various parameters to produce nanoparticles.

Salts	Experiments / batch	Time / experiment. (In minutes)	Weight in grams
NaCl	06	11 - 14	50 - 70
KCl	06	7 - 10	100 - 120
LiCl	06	10 - 12	40 - 50

Table 5. refers to the time duration for conducting the experiments per batch and the amount of nanopowder procured. It has been observed that the Potassium Chloride electrolyte procures nanoparticles more effectively than the other two electrolytes in terms of time and quantity.

Table 6: Production cost based on different electrolytes.

Salts	50 grams of Magnetic nanoparticles production cost using Electric Arc - Discharge technique (In Dollars)	Vendor price in Dollars per 50 grams
NaCl	$10	$250
KCl	$14	
LiCl	$40	

It costs almost $250 to purchase 50 grams of magnetic nanoparticles from any vendor in the open market whereas the nanoparticles synthesized using Electric Arc-Discharge technique costs a maximum of $40 as shown in Table 6. LiCl salt has its own constraints because of the government-imposed rules according to human and nature concern.

CONCLUSIONS

The conclusions are as follows:

- The Electric Arc Discharge Technique is an effective method for producing magnetic nanoparticles with a diameter of less than 50 nm.
- The production rate is slower with NaCl and LiCl electrolytes compared to KCl under neutral conditions, indicating that KCl accelerates the production process.
- Previous studies have highlighted the advantages of using KCl electrolyte for producing magnetic nanoparticles, particularly in terms of cost efficiency and production rate.
- The average size of nanoparticles produced using LiCl electrolyte is smaller compared to those produced with NaCl and KCl electrolytes, while KCl electrolyte leads in terms of production quantity.

SCOPE FOR FUTURE WORK

The Electric Arc Discharge Technique was operated at a constant voltage of 70 V. By varying the voltage according to the specifications of the equipment, a broad range of magnetic nanoparticles can be produced, including those with additional magnetic elements like nickel and cobalt, allowing for diverse applications. Future experiments can be optimized to achieve more uniform magnetic nanoparticles.

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