

Influence of Electrolyte on the Size of Magnetic Iron Oxide Nanoparticles Produced Using Arc-Discharge Technique

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Abstract - Magnetite and hematite were produced in ultra-fine nanoparticles form using the arc-discharge method. This magnetic nanoparticle finds many applications, including magnetic resonance imaging, memory disk, magnetic gel, ferrofluids, and magnetic refrigeration in addition to many other chemical and medical applications. Therefore, many efforts are ongoing all over the globe to identify the ways and means to come out with an economical and uncomplicated method. Among all those methods one of the low-cost methods is the arc-discharge method to produce ultra-fine magnetic nanoparticles using electrolytes like NaCl, KCl, and LiCl salts which are easily and cheaply available. The magnetic properties of the nanoparticles obtained are characterized by the V.S.M technique. XRD technique is used for phase identification, sample purity, and particle size. The samples are also characterized by S.E.M to know the morphology of microstructure. A particle analyzer is also used to estimate the particle size. This study shows clearly the relationship between various processing parameters and magnetic properties.

Keywords – Characterization, Ferrofluids, Magnetic nanoparticles, Scanning electron microscope, Vibrating sample magnetometer, X-ray diffraction.

INTRODUCTION

Nanoscience is one-off the prominent areas, which plays a vital role in diverse research fields. Nanotechnology has attracted many engineers, scientists, chemists to work at the molecular level to give rise to new technologies, which help to improve the overall life of human beings by ways of utilizing them in different devices and systems. Among nanomaterials, magnetic nanoparticles find countless applications in various fields. These magnetic nanoparticles are used in ferrofluids as an active component [1], magnetic refrigeration [2,3], catalysis [4,5], biomedicine [6], cancer therapy [7,8], magnetic resonance imaging (MRI) [9,10, 11], waste-water treatment [12], nanofluid hyperthermia [13], data storing devices [14], microwave absorption [15], and medical diagnosis [16]. Magnetite and hematite nanoparticles are extensively used in numerous applications, due to their easy availability and superparamagnetic properties [17]. The research on nanoparticles has given much importance due to their attractive and superior chemical and physical properties over their bulk cousins. Many techniques were developed in producing magnetic nanoparticles like co-precipitation [18], hydrothermal treatment [19], sol-gel method [20], Decomposition of gas-phase method [21], Electron beam lithography technique [22], Sonochemical method [23], Electrochemical technique [24], and Microbial incubation technique [25]. Apart from advantages, there are some disadvantages or limitations in each method mentioned above. The co-precipitation method is cost-effective but the magnetic nanoparticles get easily oxidized therefore it requires nitrogen for protection from oxidation. The hydrothermal treatment method has great potential applications but requires special tools and apparatus by which the production cost relatively increases. In this method, the pressure and reaction temperature required is also high; therefore, this method cannot be industrially used. Magnetic nanoparticles obtained using the hydrothermal method are mostly hydrophobic, which greatly limits the applications. In the sol-gel method, the nanoparticles obtained are pure, also a low-cost method but nanoparticles morphology, size cannot be completely controlled. Since many chemical processes are involved in the sol-gel method due to which these chemicals are considered to be toxic and which will affect the environment, therefore this method is not considered to be eco-friendly. Decomposition of the gas phase is one of the physical methods which can be easily executed but the nanoparticle size cannot be well-controlled. In the electron beam lithography method, interparticle spacing is

well maintained but it requires costly and complex tools to operate. Sono-chemical decomposition method and electrochemical methods are both chemical methods where particle size is controllable but requires costly complex tools, and the mechanism is very complicated. Also, it cannot reproduce nanoparticles. Microbial incubation is a biological method that produces nanoparticles on large scale and at a low cost but the production process is very slow and requires huge labor. To overcome the limitations and also to prepare magnetic nanoparticles in large quantity that can be used on an industrial scale, hence arc-discharge method [26], is used in making magnetic nanoparticles economically, using simple equipment and procedure. When differentiated with other methods, this method produces magnetic nanoparticles in large quantities. In this paper, the synthesis of magnetic nanoparticles using NaCl, KCl, and LiCl electrolytes is reported. The synthesis using KCl and LiCl electrolytes, to our knowledge was not reported previously. Knowledge of the size and shape of nanoparticles is necessary to know the magnetic, catalytic, and optical characters of the material for practical use. It is also known that solid matter consists of both amorphous and crystalline structures [27]. Inspection based on agglomeration [28] and oxidation of the particles is very important while calculating the mean size of nanoparticles [29]. However, these are very sensitive to agglomeration and oxidation [2]. This agglomeration can be reduced by various coatings such as silica and zinc etc. Additionally, this coating reduces oxidation and enhances the properties of nanoparticles [30, 31].

EXPERIMENTAL PROCEDURE



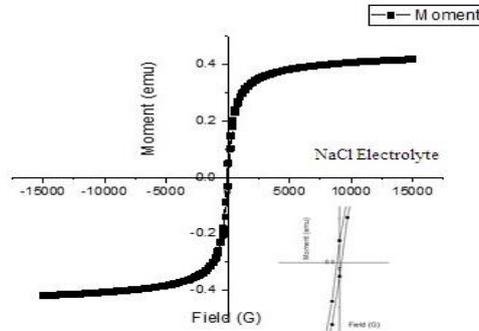
FIGURE 1
EXPERIMENTAL SETUP

In this method, magnetic nanoparticles were generated using the arc-discharge method [26]. The diameter of the electrodes (ER70S-2) used for both anode and cathode is 1.6mm. The chemical composition (wt%) of the electrode used is as follows, C-0.05%, Cu-0.35%, Mn-1.15%, Ti-0.06%, Si-0.45%, Zr-0.04%, P-0.012%, Al-0.09%, S-0.012%. Before the experiment, the electrodes were cleaned using Nital (etching) until the complete surface of the electrode gets free from the copper coating. An aqueous solution is formed by mixing individual salts based on their respective molecular weight (NaCl-58.44gm/mole, KCl-74.55gm/mole, LiCl- 42.39gm/mole) with distilled water. Then the solution is poured into a 1500ml beaker. Now the electrodes were placed in the solution by maintaining a gap of 3cm between the electrodes and a depth of 6cm inside the solution, to get the required arc. During the electrolysis process, a reaction that takes place between the electrolyte and electrode is rigorous that causes more heat. Hence the beaker is surrounded by ice to prevent any explosion that might occur due to the heat produced. The essential setup is shown in Fig. 1. A 50Hz alternating current with a varying voltage of (20-100) V is used in this experiment. Previously using NaCl electrolyte, the experiment was conducted by C.Y. WANG [26] using electrodes other than ER70S-2 but for the first time, we use KCl and LiCl electrolytes to synthesize magnetic nanoparticles. During the experiments, a strongly exothermic reaction takes place, and suddenly the electrodes start dissolving and leave black and brown precipitates in the electrolyte. Iron colloids are formed in the solution due to these strong exothermic reactions. These active iron colloids were then oxidized to Fe^{2+} , later forms $Fe(OH)_2$ in the electrolyte, and also oxidizes to $Fe(OH)_3$. Since the temperature recorded near the electrodes is high which results in oxidation [2]. The experiment was conducted only for 15 minutes because by this time the reaction rate slows down. The magnetic nanoparticles obtained were separated from the solution by using filter paper. Now the magnetic nanoparticles thus obtained were washed using water 3 to 4 times and later with absolute alcohol to eliminate the presence of salt which was added in electrolysis. After completion of this process, the nanoparticles were heat-treated at 80°C for 15 hours in a heating furnace. The heat treatment given to the nanoparticles helps to dry up the moisture content from the nanoparticles and also to obtain the required crystal structure of the material. Now the magnetic nanoparticles thus obtained were stored in an airtight container to get rid of further oxidation. The study of electrolysis based on different electrolytes, fluctuations in current, diametric changes (ϕ) before and after the experiment are noted.

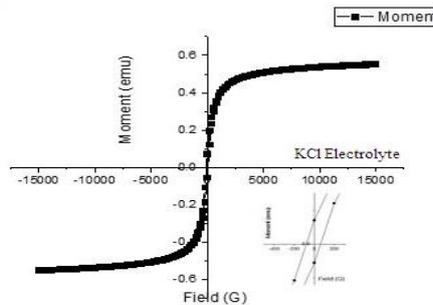
RESULTS

1. VSM Results of Magnetic Nanoparticles Using NaCl, KCl, and LiCl Electrolytes at 70V

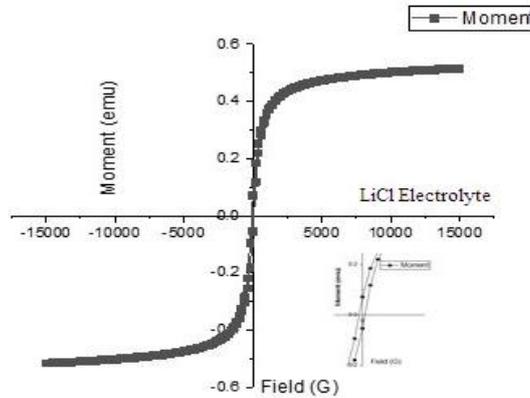
Vibrating Sample magnetometer [32] is an inductive method by which the magnetic properties of materials can be known. A hysteresis graph between moment (emu) and applied field (G). The parameters which are identified from the graph are the magnetic remanence (Mr), saturation magnetization (Ms), the coercivity (Hc), the squareness ratio (SQR). The application of magnetic nanoparticles is based on SQR i.e., in memory storing devices. The SQR value should be as large as possible whereas in the magnetic fluid it needs to be as low as possible. If the SQR value is less than 0.5, then it indicates a small single domain.



GRAPH 1
HYSTERESIS CURVE OF MAGNETIC NANOPARTICLES USING NaCl ELECTROLYTE



GRAPH 2
HYSTERESIS CURVE OF MAGNETIC NANOPARTICLES USING KCl ELECTROLYTE



GRAPH 3
HYSTERESIS CURVE OF MAGNETIC NANOPARTICLES USING LiCl ELECTROLYTE

TABLE I
MAGNETIC PROPERTIES OF MAGNETIC NANOPARTICLES USING NaCl, KCl, LiCl ELECTROLYTES

Parameters	NaCl	KCl	LiCl
Mr	0.04949	0.07086	0.07047
Ms	0.41937	0.55302	0.51470
Hc	46.50	63.05	62.54
SQR	0.11	0.12	0.13

2. XRD Result Analysis

XRD calculation of magnetic nanoparticles under NaCl, KCl, and LiCl Electrolytes before and after heat treatment process at 70V.

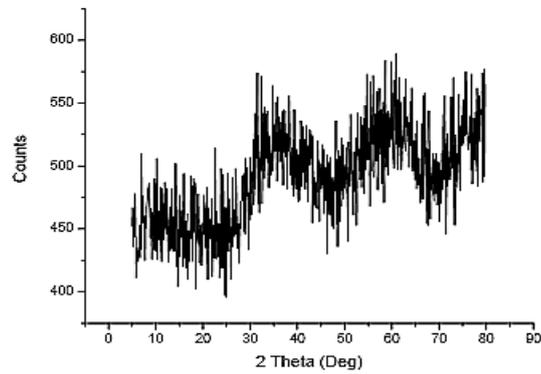


FIGURE 2-A
XRD PATTERN OF ER70S-2 IN NaCl ELECTROLYTE

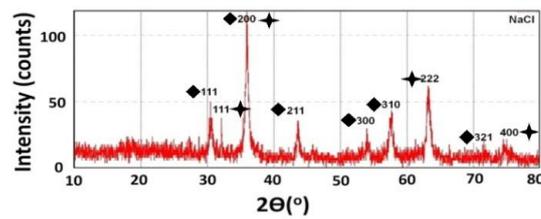


FIGURE 2-B
XRD PATTERN OF ER70S-2 IN NaCl ELECTROLYTE

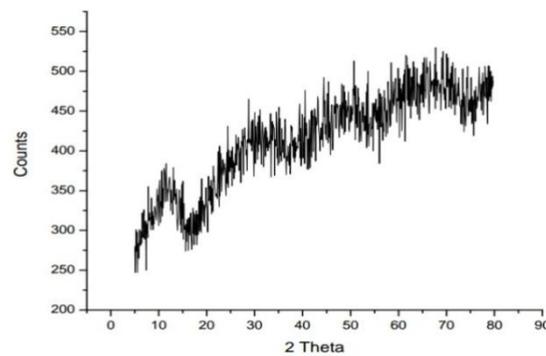


FIGURE 3-A
XRD PATTERN OF ER70S-2 IN KCl ELECTROLYTE

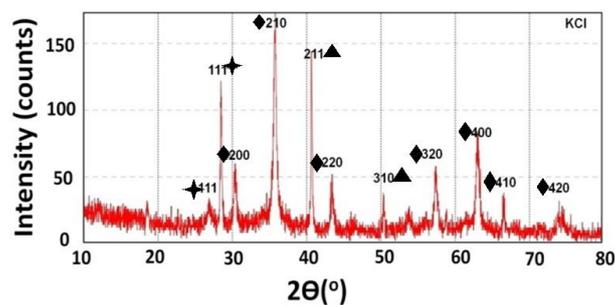


FIGURE 3-B
XRD PATTERN OF ER70S-2 IN KCl ELECTROLYTE

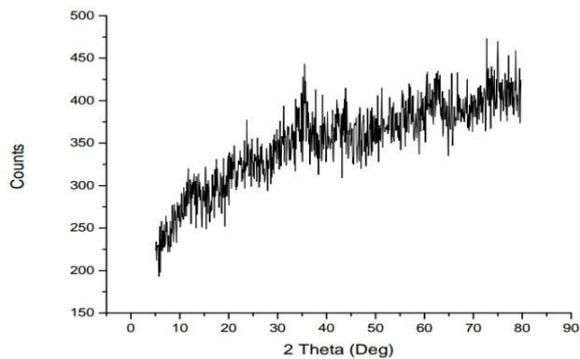


FIGURE 4-A
XRD PATTERN OF ER70S-2 IN LiCl ELECTROLYTE

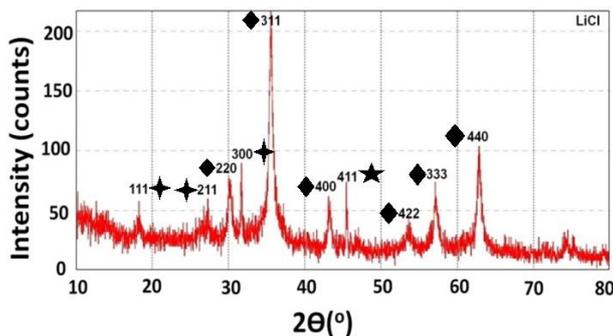


FIGURE 4-B
XRD PATTERN OF ER70S-2 IN LiCl ELECTROLYTE

The figures mentioned above (2-a, 3-a, and 4-a) represent the XRD [18] patterns of ER70S-2 in different electrolytes before heat treatment. We can notice that there are no strong peaks in the figures which represent that the rays passing through the material did not recognize the material properties, which indicates the structure is amorphous [33]. Similarly, Figures (2-b,3-b, and 4-b) represent the XRD patterns of ER70S-2 in different electrolytes after heat treatment. We can notice that there are many strong peaks obtained which represent that the rays passing from the material did recognize the properties of the nanoparticle which indicate the structure as crystalline [34]. Based on the 2θ values, the element name can be identified. Based on the diffraction angle (2θ) element name can be identified from table-3. In the XRD pattern of ER70S-2 peaks are identified as Fe_3O_4 and Fe_2O_3 based on their 2θ values [9,29]. Here $2\theta=35.53^\circ$ is a standard diffraction angle of Fe_3O_4 nanoparticle with cubic structure [30, 35]. The traces of iron oxide and iron were also identified in the XRD report.

TABLE 2
STANDARD 2θ VALUES OF MAGNETIC NANOPARTICLES

PHASE NAME	2θ (degrees)
Fe_2O_3	17.90, 27.20, 33.20, 35.53, 38.93, 45.20, 54.20, 63.80 and 75.30
Fe_3O_4	30.16, 35.53, 43.18, 53.40, 57.11, 62.71, 66.20, 71.00, 74.40
Fe	45.30
FeO	41.20, 50.80

3. Particle Size Calculation

The mean particle size of the nanoparticles which were in powder form was estimated using the Debye-Scherrer equation [36, 37]. Accordingly,

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

Here, D = Particle size in nm.

λ = Wavelength (0.154 nm).

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

θ = Bragg's Diffraction angle.

TABLE 3
PARTICLES SIZE OBTAINED USING DEBYE-SCHERRER FORMULA

Electrolyte	Minimum size(nm)	Maximum Size(nm)	Average size(nm)
NaCl	17.32	31.06	22.52
KCl	19.94	35.44	27.99
LiCl	14.48	28.78	20.85

SEM RESULTS

An SEM (S-3700N) with accelerating voltage varying between 0.3-30kV gives the required information in the form of an image of the sample by scanning the surface of the magnetic nanoparticles [38]. The minimum and maximum size of the nanoparticles obtained using SEM at 70V is shown in Table 4. Before conducting S.E.M analysis, an Ultrasonic cleaning bath is done on these magnetic nanoparticles which creates dispersion and reduces agglomeration. The ultrasonic cleaner also reduces the size of the nanoparticles which results in more uniform particle size. Agglomeration is the foremost reason which may bring changes in the SEM results when compared with other results, so precautions are to be taken to obtain the size of the actual nanoparticles. The SEM image results as shown in Fig 5, 6, and 7 represent the obtained nanoparticles sizes.

SEM images of magnetic nanoparticles under NaCl, KCl, and LiCl Electrolytes at 70V.

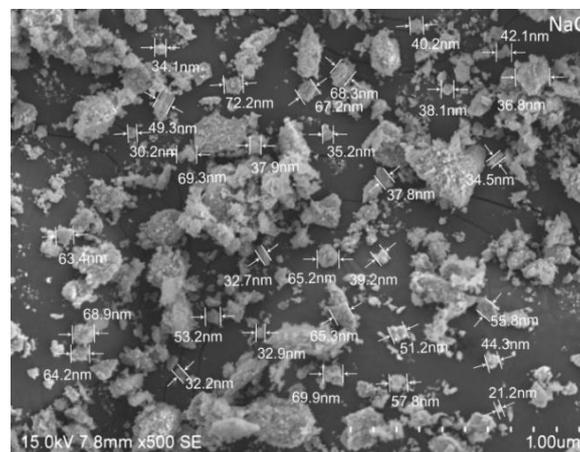


FIGURE 5
SEM OF ER70S-2 USING NaCl ELECTROLYTE

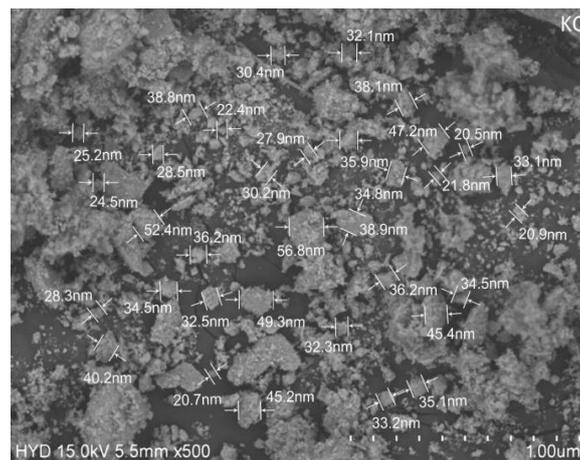


FIGURE 6
SEM OF ER70S-2 USING KCl ELECTROLYTE

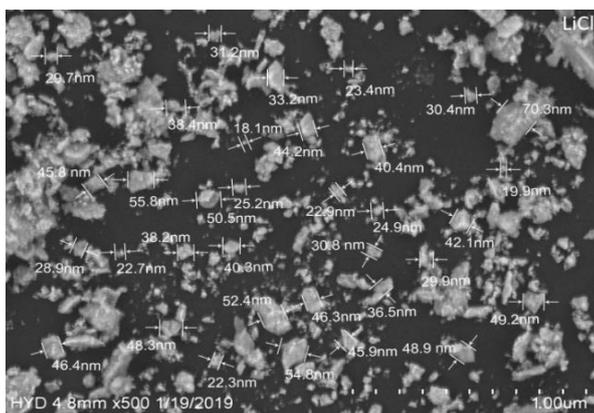


FIGURE 7
SEM OF ER70S-2 USING LiCl ELECTROLYTE

TABLE 4
MINIMUM AND MAXIMUM SIZE OF MAGNETIC NANOPARTICLES IN NM USING SEM

Electrolyte	Minimum size (nm)	Maximum size (nm)	Average size (nm)
NaCl	16.6	36.7	26.6
KCl	17.3	28.3	22.8
LiCl	16.4	32.5	24.4

PARTICLE ANALYZER RESULTS OF MAGNETIC NANOPARTICLES USING NaCl, KCl, AND LiCl ELECTROLYTES

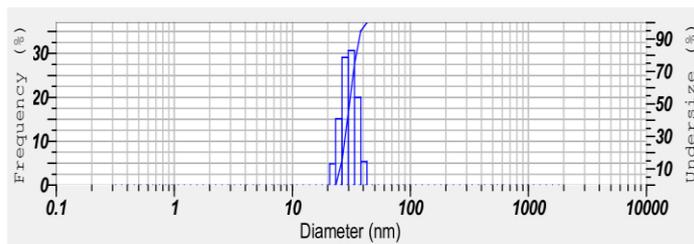


FIGURE 8
PARTICLE ANALYZER IMAGE OF MAGNETIC NANOPARTICLES USING NaCl ELECTROLYTE

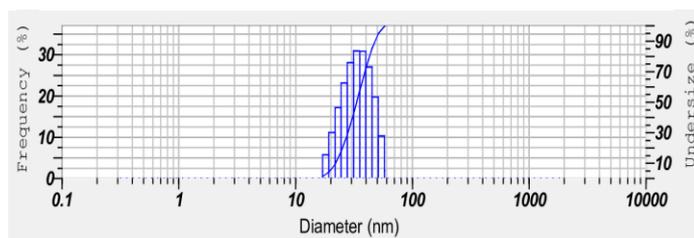


FIGURE 9
PARTICLE ANALYZER IMAGE OF MAGNETIC NANOPARTICLES USING KCl ELECTROLYTE

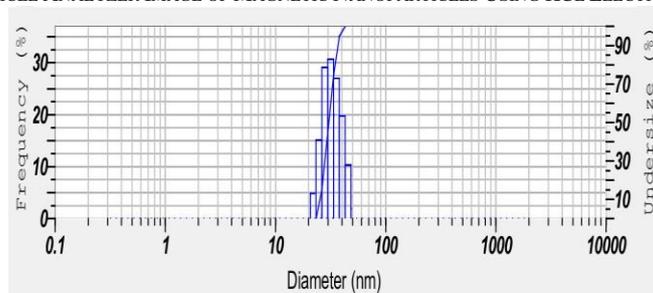


FIGURE 10
PARTICLE ANALYZER IMAGE OF MAGNETIC NANOPARTICLES USING LiCl ELECTROLYTE

TABLE 5

MINIMUM AND MAXIMUM SIZE OF NANOPARTICLES IN NM USING PARTICLE ANALYZER

Electrolyte	Minimum size (nm)	Maximum size(nm)	Average size(nm)
NaCl	20	40	30
KCl	18	50	34
LiCl	20	50	35

The results based on particle analyzer are also useful to conclude the size of the magnetic nanoparticles. Figures (8-10) represent the diametric size distribution of magnetic nanoparticles by particle analyzer. The average nanoparticle size obtained using NaCl, KCl, and LiCl electrolyte solution are 30nm, 34nm, and 35nm respectively [39, 40], and this variation in size of nanoparticles was due to agglomeration. Since the magnetic nanoparticles are immersed in the testing solution, there will be a variation in the size of the magnetic nanoparticles when differentiated with the size obtained in dry state XRD results. The size of the nanoparticles was ranged from 30nm to 35nm. When compared with the results of XRD and SEM the obtained results of the particle size analyzer are very similar

THE VARIATION IN THE DIAMETER OF THE ELECTRODE (ER70S-2) BEFORE AND AFTER EXPERIMENTING 70V IN 15 MIN

TABLE 6
VARIATION IN DIAMETER OF THE ELECTRODE USING DIFFERENT ELECTROLYTES

Electrolyte	Time (minutes)	Before Experiment	After Experiment
NaCl	15	1.6 mm	1.3 mm
KCl	15	1.6 mm	1.0 mm
LiCl	15	1.6 mm	1.4 mm

While comparing the diameters of the electrodes before and after the experiment, we can conclude that the diameter of the electrode is much reduced in KCl electrolyte as it has a high tendency to react with the electrodes. KCl electrolyte also produces magnetic nanoparticles in more quantity when compared with LiCl and NaCl electrolytes.

THE VARIATION IN CURRENT DENSITY VS TIME OF MAGNETIC NANOPARTICLES USING NaCl, KCl, AND LiCl ELECTROLYTES

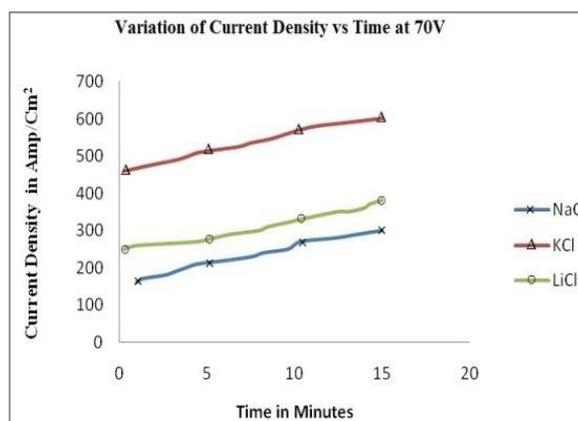


FIGURE 11
CURRENT DENSITY VS TIME AT 70V

A graph is plotted between current density and time at 70V of magnetic nanoparticles using NaCl, KCl, and LiCl electrolytes respectively. Based on the obtained graphical data, we can conclude that the current density required in KCl electrolyte is more when differentiated with LiCl and NaCl electrolytes.

DISCUSSIONS

During the experimental procedure, some important observations were made. The nanoparticles will remain in their amorphous form as produced which needs a heat treatment for conversion into a crystalline state. The role of the electrode diameter is very crucial in the synthesis of nanoparticles. As the diameter of the electrode is increased, more current is drawn. Comparatively more heat was involved in the solution while using a 2mm diameter electrode. The solution turns reddish if kept unfiltered immediately after the experiment. It was observed that after a delay in experimenting for a day or so after mixing salt in water, no powder could be produced. Almost 12-15 minutes is the time duration for one complete experiment. Nearly 15 hours of heating is required to dry the samples precipitate. In these experiments, we have observed that a smaller diameter electrode (1.6mm) gave more

quantity of powder than with a 2mm diameter electrode. When voltage is kept constant, initially the current increases and after a certain time current decreases resulting in no production of nanopowder. Our study was an attempt to know the influence of various electrolytes, voltage, current, and size of the electrodes on the synthesis of nanopowder which adds to our understanding much deeper.

CONCLUSION

Magnetic Fe₂O₃ and Fe₃O₄ nanoparticles were prepared successfully using the arc-discharge technique. The results of VSM, XRD, SEM, and Particle analyzer show that the pure magnetic Fe₂O₃ and Fe₃O₄ nanoparticles were synthesized with different processing parameters like variation in voltage and current and also by maintaining the distance between the electrodes. VSM patterns show the SQR values less than 0.5 which indicates a small single domain. The formation of iron-based nanoparticles is represented by XRD patterns. From the results obtained, we conclude that the size of the nanoparticles calculated was less than 35nm in XRD and confirmed by SEM and particle analyzer methods in various electrolytes (NaCl, KCl, and LiCl). A very small difference in the size of magnetic nanoparticles in the XRD pattern, S.E.M photographs, and particle analyzer graphs indicate the agglomeration of particles. When compared with LiCl and NaCl salts, the KCl electrolyte is highly beneficial as it allowed producing more quantity of nanoparticles. This study indicates the benefits of various electrolytes and various processing parameters in the arc-discharge method.

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All authors declare no conflicts of interest in this paper.

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