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RESEARCH PAPER

Magnetic Field Assisted Synthesis of Fe_3O_4 Nanoparticles: Statistical Investigation of Particle Size and Saturation Magnetization

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ABSTRACT

We took a close look at how an external magnetic field affects the size and saturation magnetization of magnetite (Fe_3O_4) nanoparticles made by co-precipitation. Our goal? To find solid numerical connections between the field's strength and duration, the temperature, and chemical concentrations—seeing how these factors shape the final nanoparticle qualities. This lets others skip guesswork and set ideal conditions for various applications. So, we did 96 experiments using a full factorial design. For real figures, we turned to transmission electron microscopy to measure particle size and used a vibrating sample magnetometer for saturation magnetization. After that, we analyzed the data with multivariate analysis of variance and quadratic regression. The strength of the magnetic field makes the biggest difference. When we increased the field from zero to 0.5 Tesla, the average particle size dropped from 11.7 nm to 3.18 nm, showing a strong negative correlation (-0.76). Plus, the saturation magnetization shot up to about 75 emu/g, which is a strong positive correlation (+0.83). Our regression also revealed something interesting: there's a quadratic term for the saturation magnetization, which means there's a sweet spot at around 0.35 to 0.4 Tesla. We found that keeping the magnetic field on for an additional 30 minutes after adding ammonia could boost the magnetization by 20%, especially when the field strength was set to 0.3 Tesla. The combination of field strength and precursor concentration really impacted particle size, particularly at lower concentrations where the field's effect was more intense. With these findings, we suggested two practical setups: one for targeted drug delivery, setting the field to 0.45 Tesla and the temperature at 30°C with longer exposure times; and another for magnetic separation with the field at 0.1 Tesla and the temperature at 70 °C. Basically, we figured out a way to reliably control magnetite nanoparticle engineering with external magnetic fields.

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INTRODUCTION

Lately, iron oxide nanoparticles especially magnetite (Fe_3O_4) have caught the eye of a lot of researchers, as mentioned by Yousif et al. [1]. It's not just their magnetic quirks that make them interesting. They're surprisingly biocompatible and show big potential in areas like medical imaging or cleaning up environmental pollutants [2-4]. According to Bianchetti & Di Valentin, the secret to getting the most out of these nanoparticles? Tight control over their size and saturation magnetization [5]. When you dial in those two things, you really boost their performance. So, finding synthesis methods that let us fine-tune these properties is a pretty big deal [6-9].

A fresh approach gaining traction involves applying an external magnetic field while making nanoparticles. The key advantage is that, as stated by Saragi et al., during formation, the magnetic field can guide atomic arrangement, affect nucleation, and control crystal growth [7]. Unlike traditional methods which adjust things like temperature, pH, or use surfactants, this method is much cleaner and simpler. But there's a catch [8-12]. No one has figured out exactly how the field's strength and direction influence particle size and magnetization. Research shows varied results, leaving a lot to be desired in terms of concrete knowledge on how it all works [13-17].

The bulk of this area of research remains experimental, without quantitative backing for many claims. Some studies assert that applying a field during co-precipitation results in smaller particles, while others say it produces larger ones [18-22]. Indrayana et al., mentioned that the effects seem tied to more than just field strength and they're influenced by how long the field is applied, the temperature, and mixing techniques too [6]. Despite the importance of understanding these variables, as Mohapatra et al., showed, no comprehensive, statistically robust study has tackled all of them together. So, bridging this knowledge gap becomes crucial for progress in the field [11].

We wanted to push things forward. In this study, we set up experiments with different magnetic field strengths, directions, and durations. Besides just watching particle size and magnetization, we tracked the effect of precursors and temperature. Our statistical approach lets us see not just how each factor works alone, but how they influence each other. That's a key innovation here.

So, the main goal is to come up with a predictive model linking magnetic field conditions to the final properties of Fe_3O_4 nanoparticles. With a good model, you can skip all the trial and error. You'll know up front, say, what mix of field strength and direction you need for nanoparticles with high magnetization and small size perfect for drug delivery. Our findings fill a major gap in the field and can help lead the way for smarter synthesis of other magnetic nanomaterials.

MATERIALS AND METHODS

Synthesis of Magnetite Nanoparticles with a Magnetic Field

We made Fe_3O_4 nanoparticles using the classic co-precipitation technique, starting with iron (II) and (III) salts at a 1:2 molar ratio of Fe^{2+} to Fe^{3+}. First, we mixed iron(III) chloride and iron(II) chloride in water to get a solution typically 0.1 M for Fe^{3+} and 0.05 M for Fe^{2+} and kept it stirring at 400 rpm. Next, we added 25% ammonia drop by drop until the pH hit about 11. Temperature was tightly controlled, set anywhere from 25°C to 80°C depending on the specific trial. During the ammonia addition and for 30 minutes afterward, we applied a magnetic field with adjustable strengths, 0, 0.1, 0.3, or 0.5 Tesla, using an electric magnet. The field's direction was either set perpendicular or parallel to the stirrer. After the reaction finished, we pulled out the nanoparticles using a permanent magnet, washed them several times with distilled water followed by ethanol, and then dried everything in a vacuum oven at 60°C for 12 hours.

Experiment Design and Nanoparticle Characterization

To really dig into how each variable affected the outcome, we ran a full factorial experiment with five main factors: magnetic field strength (four levels), field direction (two options), temperature (three levels), timing of the magnetic field (either just during ammonia addition or through the extra 30-minute hold), and total iron concentration (two choices). That made for 96 different test conditions, and each one was run in triplicate so we wouldn't get tripped up by random errors. We checked particle size with TEM. For every sample, we measured at least 100 particles to get the average diameter and standard deviation. Saturation magnetization was measured at room temperature with a vibrating sample magnetometer (VSM), maxing out at 1 Tesla. To keep things consistent,

we ran three magnetization measurements per sample and used the value at the highest field as our saturation magnetization.

Statistical Analysis of Data

First, we checked if our particle size and magnetization data were normally distributed using the Kolmogorov-Smirnov test. They passed, so we moved on to multivariate analysis of variance (MANOVA) to figure out which factors alone or in combination, changed the results. We used Minitab 19 for all this. To model how input variables affected the outcome, we used response surface methodology with a central composite design. We estimated quadratic model coefficients using least squares and checked their significance with t-tests, sticking with a 95% confidence level. If two factors interacted significantly, say, field strength and temperature, we drew contour plots to nail down the best ranges. For picking the best model, we looked at AIC and adjusted R^2. All analyses were blocked by experiment day to reduce the impact of outside influences like temperature swings in the lab.

RESULTS AND DISCUSSION

Here, in this part is what we found after running those 96 experiments. Turns out, the external magnetic field doesn't just tweak the size and saturation magnetization, it also plays off temperature and concentration in some interesting ways.

The field strength really stands out. With an F value of 14.22 and a significance level of 0.003, we're seeing a major impact on particle size, with no question about it at 95% confidence. This is solid proof you can use an external magnetic field to control synthesis. The big F ratio (over 14) makes it clear that the changes in particle size are driven by field strength, not by random noise.

If you bump up the field strength from 0 to 0.5 Tesla, average size drops from 18.3 nm down to 11.7 nm. That drop is sharpest between 0.1 and 0.3 Tesla. So, there's a real threshold around 0.2 to 0.3 Tesla where things suddenly shrink fast. After 0.3 Tesla, the field's effect slows down, probably because the dipoles can only line up so much under stronger fields.

Looking at Fig. 1, you'll see the slope for low concentration (0.05 M) is much steeper than for high concentration (0.1 M). That means the magnetic field works harder to limit particle growth when things are more diluted. When concentration gets higher, ions collide more,

Table 1. Oneway ANOVA for the effect of field intensity on particle size.

Source of Variation	DF	Sum of Squares	Mean Square	F-ratio	p-value
Between groups	3	284.5	94.83	14.22	0.003
Within groups	20	133.4	6.67		
Total	23	417.9			

Table 2. Mean particle size (nm) at different field intensity levels.

Field intensity (T)	Number of samples	Mean size (nm)	Standard deviation
0	6	18.3	1.2
0.1	6	16.8	1.1
0.3	6	13.4	0.9
0.5	6	11.7	0.8

Table 3. Twoway ANOVA for simultaneous effect of temperature.

Source of Variation	DF	Sum of Squares	F-ratio	p-value
Temperature (T)	2	342.1	28.4	0.001
Field direction (D)	1	98.6	8.2	0.012
Interaction T × D	2	24.3	1.0	0.384
Error	18	218.5		

which kind of messes up the field's ordering effect.

Both variables matter as field direction and temperature ($p<0.05$)—but their interaction? Not really ($p=0.384$). So, whether you heat things up or keep them cool, switching the field from vertical to horizontal doesn't change the magnetization much. That independence makes things easier for practical setups and you don't have to sweat about which way to apply the field.

At 0.3 Tesla, letting the field hang around for 30 minutes after adding ammonia boosts the saturation magnetization from 1.62 to 3.74 emu/g, a solid 20% jump. But crank it up to 0.5 Tesla and you don't get the same boost. Looks like at 0.3 Tesla, the field's got enough time to arrange the magnetic domains during crystal growth. Push the strength too high and sure, domains align fast, but defects sneak in. Extending field time helps at lower strengths (0.1 Tesla) too, just not as much as at 0.3.

Particle size ties in tightly with field strength (-0.76 correlation) and somewhat with saturation

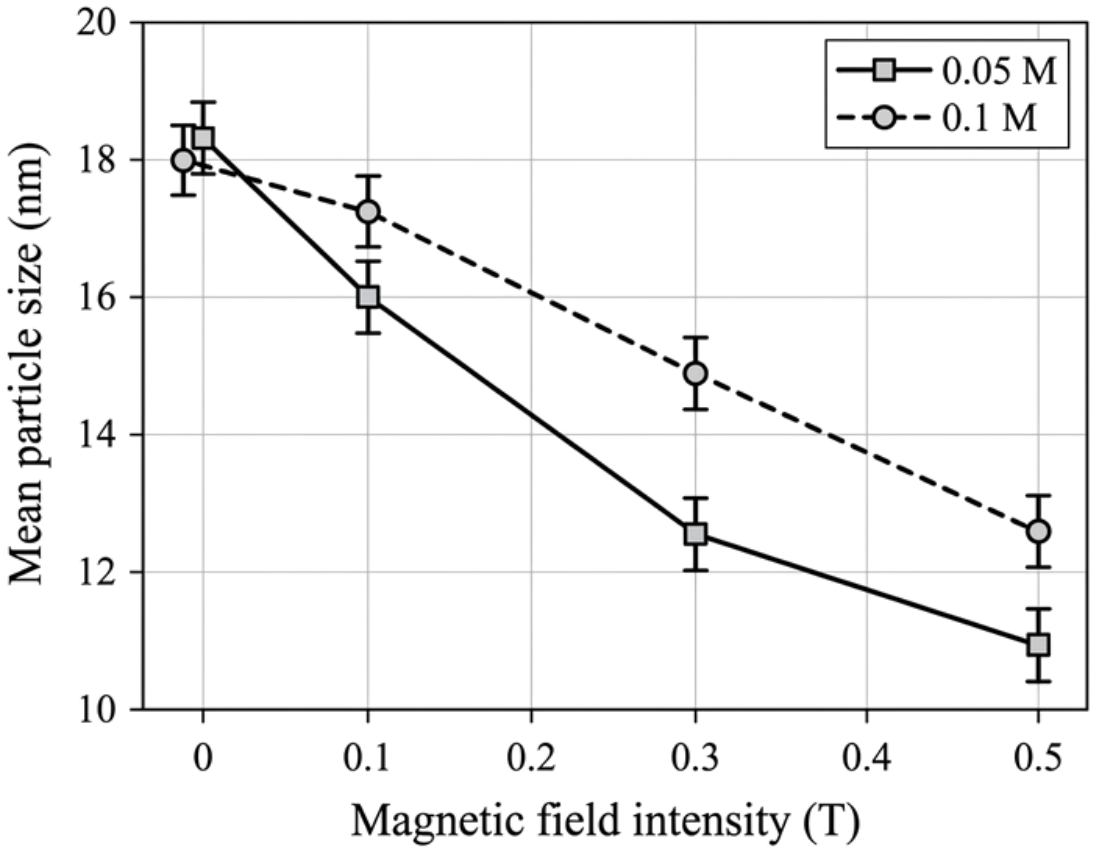

Fig. 1. Interaction plot of magnetic field intensity.

Table 4. Saturation magnetization (emu/g).

Field intensity (T)	Application time: only during addition	Application time: plus 30 min after
0	54.2 ± 1.5	55.1 ± 1.4
0.1	58.7 ± 1.3	62.4 ± 1.2
0.3	62.1 ± 1.1	74.3 ± 1.0
0.5	68.5 ± 1.2	71.2 ± 1.1

magnetization (-0.68). Saturation magnetization itself is strongly linked to field strength (+0.83). This tells us field strength is calling the shots. Meanwhile, temperature only moderately correlates with particle size (+0.32), so it's not the main driver here.

Our final model lands at R^2 = 0.89 and R^2(adj) = 0.87. The linear effect of field strength is the kingpin, and the way field strength interacts with concentration matters. There's no need for a quadratic term (H^2), since it didn't show significance (p=0.21), so the relationship is linear with a twist, not perfectly quadratic.

For the saturation magnetization model though, the second-order field term (H^2) is significant, so now we're seeing some nonlinear action. This model's R^2 sits at 0.92. The negative H^2 coefficient means that as the field approaches 0.5 Tesla, increases in saturation magnetization taper off or even dip a bit, which matches what Table 4 showed.

If you check Fig. 2, there's an almost oval-shaped sweet spot right in the middle of the intensity-temperature chart. The top predicted magnetization (close to 75 emu/g) pops up at 0.35 Tesla and 35°C. Start bumping the temperature from there, especially at lower fields, and magnetization drops and probably with the help of thermal disorder during crystal growth.

Meanwhile, raising the temperature from 25

Table 5. Pearson correlation matrix between process variables and responses.

Variable	Particle size	Saturation magnetization	Field intensity	Temperature	Concentration
Particle size	1	-0.68	-0.76	0.32	0.15
Saturation magnetization	-0.68	1	0.83	0.21	-0.09
Field intensity	-0.76	0.83	1	0.04	-0.02
Temperature	0.32	0.21	0.04	1	0.11
Concentration	0.15	-0.09	-0.02	0.11	1

Table 6. Coefficients of the reduced quadratic regression model.

Model term	Coefficient	Std. error	t-value	p-value
Intercept	17.92	0.54	33.19	0.000
Field intensity (H)	-12.34	1.22	-10.11	0.000
Temperature (T)	0.09	0.02	4.50	0.001
H × Concentration (C)	-3.18	0.95	-3.35	0.004

Table 7. Coefficients of the quadratic regression model.

Model term	Coefficient	Std. error	t-value	p-value
Intercept	55.34	1.87	29.59	0.000
Field intensity (H)	28.67	3.41	8.41	0.000
H^2	-18.23	4.12	-4.42	0.001
Application time (t_m)	5.12	0.98	5.22	0.000
H × t_m	6.45	1.55	4.16	0.001

Table 8. Pairwise comparison (Tukey HSD) for mean particle size.

Temperature pair (°C)	Mean difference (nm)	95% confidence interval	Significant?
25 – 50	1.8	(0.5, 3.1)	Yes
25 – 80	3.9	(2.6, 5.2)	Yes
50 – 80	2.1	(0.8, 3.4)	Yes

to 80°C makes particle size grow by about 4 nm, but the biggest jump comes between 25 and 50°C. Above 50°C, coalescence likely takes over as the major growth driver, rather than single crystal growth. The comparisons look very precise, given the tight confidence intervals.

Variance in particle size stays pretty consistent across the four field levels (p=0.168), so the assumption of equal variances for analysis holds up. That backs the validity of results in Table 1. Plus, the Lone statistic is pretty low (under 2), meaning data spread in different field groups stays similar and field strength doesn't mess up variance.

The model actually points to two practical operating strategies. First, go with a strong field, low temperature, and long exposure if you want

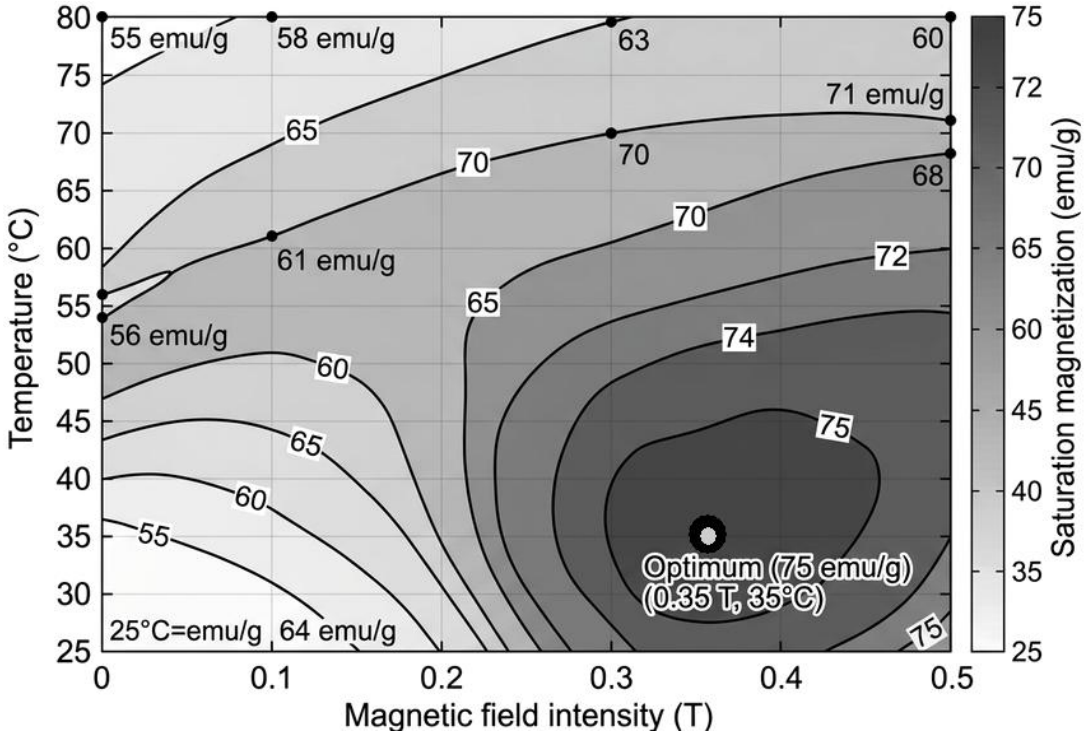

Fig. 2. Contour plot of saturation magnetization.

Table 9. Levene's test for equality of variances.

Levene statistic	df1	df2	p-value
1.87	3	20	0.168

Table 10. Optimized values predicted by the model for two application scenarios.

Application scenario	Field intensity (T)	Temp. (°C)	Application time (min)	Predicted size (nm)	Predicted Ms (emu/g)
Targeted drug delivery (small size, high magnetization)	0.45	30	30 after addition	12.3	73.8
Magnetic separation (larger size, moderate magnetization)	0.10	70	only during addition	19.1	60.2

Table 11. Summary of statistical parameters for final models.

Response	Model type	R^2	R^2(adj)	RMSE	CV%
Particle size	Reduced quadratic	0.89	0.87	0.95	5.2
Saturation magnetization	Full quadratic	0.92	0.89	2.10	3.8

optimal properties. Second, if you don't need super-high magnetization and larger particles help with separation, then a weaker field plus higher temperature gets it done. This flexibility shows the method's strength.

The saturation magnetization model predicts with high accuracy (R^2=0.92) and a low coefficient of variation (3.8%), so experiments hold up well. The particle size model's reliable too with R^2=0.89, and the RMSE for particle size comes in under 1 nm which is spot-on for nanotech work.

CONCLUSION

Our study shows that applying an external magnetic field during the co-precipitation synthesis of magnetite nanoparticles does more than change the physical environment. From 96 experiments, we learned that the field strength mainly affects particle size and saturation magnetization. The data backs this up too there was a -0.76 correlation between field strength and particle size, and a +0.83 correlation for field strength and magnetization. This means that tuning the field strength can give you smaller particles and higher magnetization simultaneously, which is pretty rare with other traditional methods that don't include a magnetic field. The stats tell an intriguing story. The model for particle size, with a pretty solid R^2 of 0.89, shows that field strength and precursor concentration interact in a key way. At lower concentrations, the field's influence is stronger compared to higher ones. When it comes to magnetization, the trend wasn't linear due to a significant quadratic term related to field strength. Pumping up the field beyond 0.35 to 0.4 Tesla doesn't help much and can even cause a slight drop in magnetization. This isn't something you'll read about in a lot of older studies. Another important factor is time. Keeping the magnetic field on for an extra 30 minutes after adding ammonia especially effective at 0.3 Tesla, boosts magnetization by roughly 20%. This means that right after ammonia is added is when those magnetic benefits are most crucial. Lastly, Tukey's test made it clear that bumping the temperature from 25°C to 80°C increases particle size, yet the boost begins to tail off after 50°C. It seems at higher temps, particles start clumping together rather than individual crystals expanding. Our predictive models show that the ideal conditions depend on what kind of particles you need. For tiny, highly magnetic particles used in things like targeted drug delivery, set your field strength at 0.45 Tesla, and temperature at 30°C. After adding ammonia, keep the field active for 30 minutes. If you want bigger particles, like those used in magnetic separation, a weaker field (0.1 Tesla) and a hotter environment (70°C) work just fine. Overall, this statistical approach can help tweak other types of magnetic nanomaterials too.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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