Synthesis of Monodisperse Magnetite Nanoparticles Using a High Pressure Homogenizer

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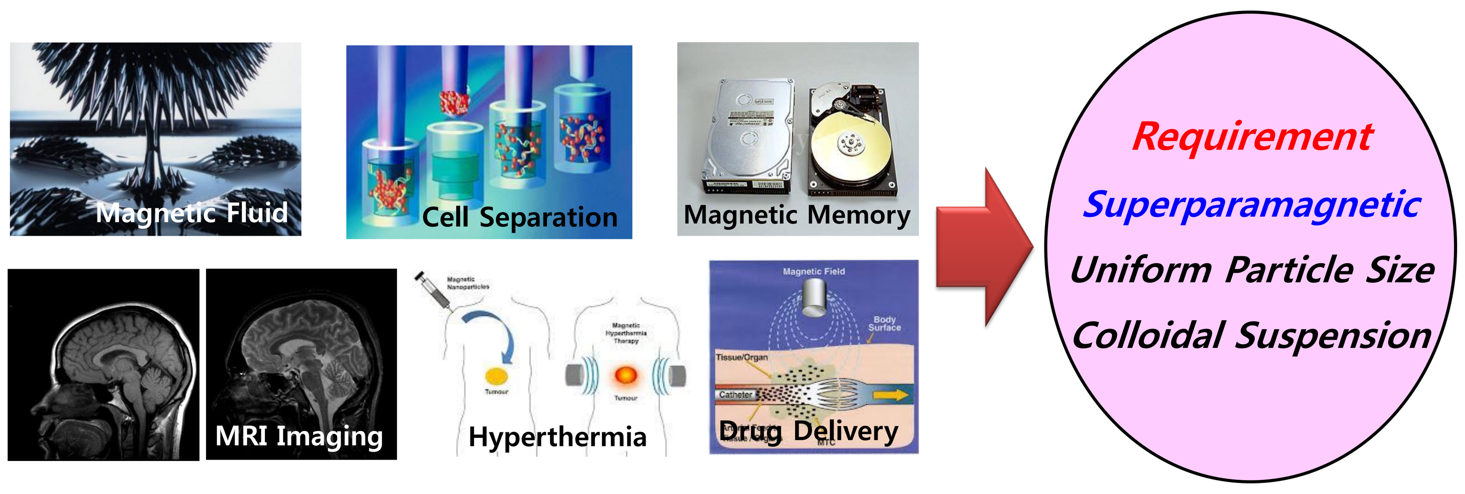
1. Introduction

➢ Applications of Magnetite Nanoparticles

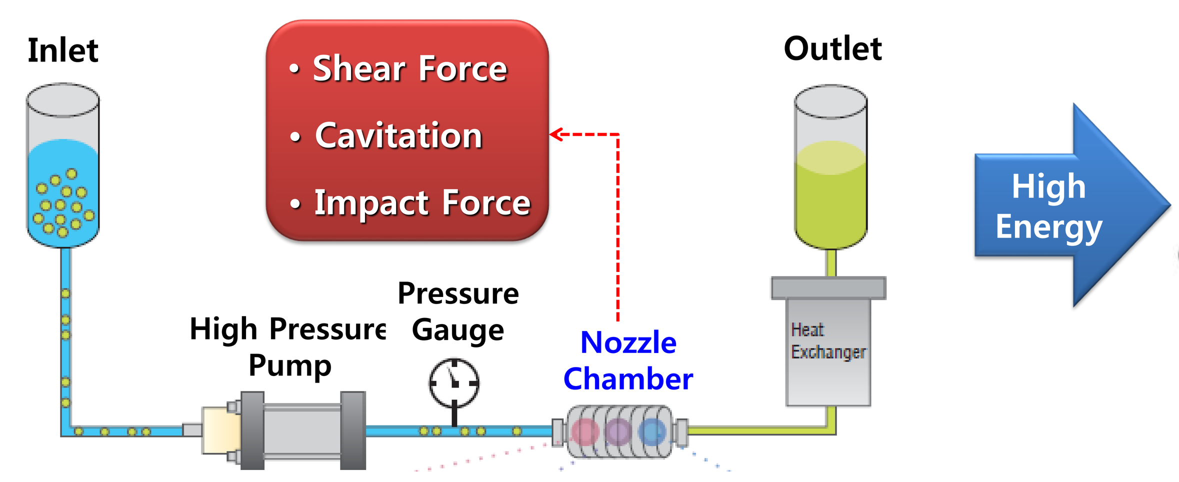


- Magnetite nanoparticles have received intensive interest in recent years due to their potential applications in various fields, such as in magnetic memory devices, magnetic fluids, magnetic refrigeration, magnetic resonance imaging and targeting drug delivery systems

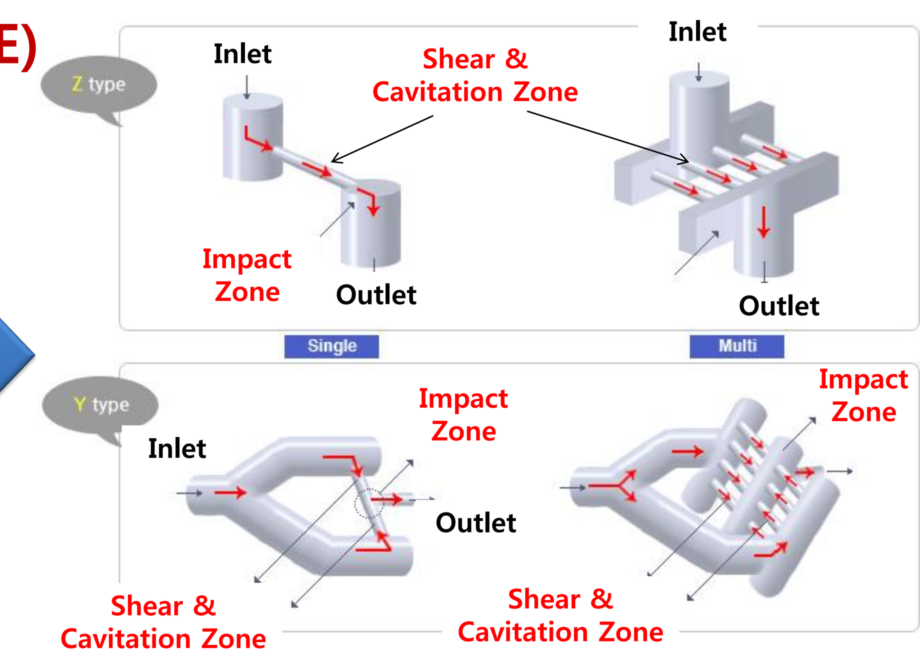
➢ Objectives of This Study

- Synthesis of uniform magnetite nanoparticles using a high pressure homogenizer without any dispersing agent.
- Control of the size of the magnetite nanoparticles.
- Synthesis of superparamagnetic magnetite nanoparticles

➢ High Pressure Homogenizer (HPH, Nano Disperser, ILSHIN AUTOCLAVE)



➢ Nozzle Chamber



➢ Fluid Velocity (Sound Speed: 340 m/s)

Pressure (bar)	500	1000	1500	2000	2500
Fluid velocity (m/sec)	313	442	542	626	700

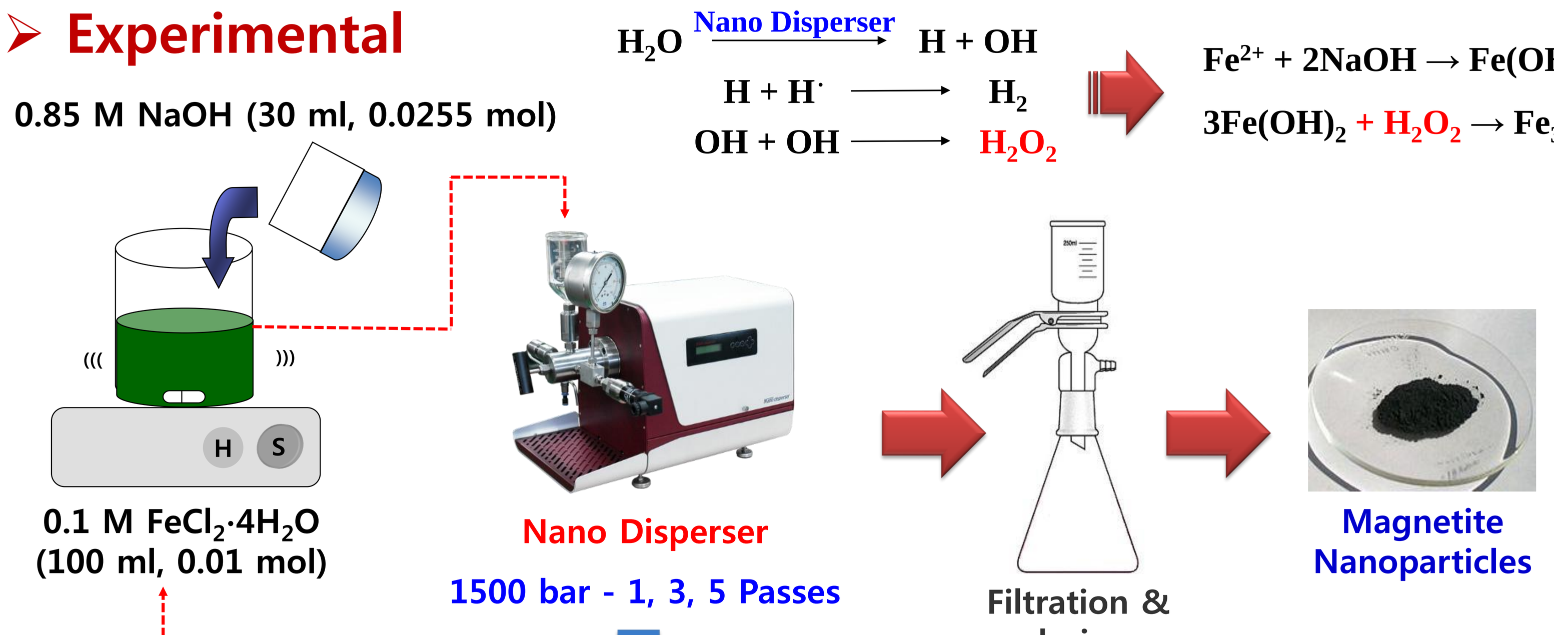
- Cavitation
  - the formation, growth, and implosive collapse of bubbles in a fluid
- Cavitational Collapse
  - intense local heating(~5000 K)
  - high pressures(~1000 atm)
  - enormous heating and cooling rates(10^{-9} K/sec)
- Advantages
  - Highly dispersion by high energy, Short process time

2. Experimental & Results

➢ Experimental

$$H_2O \xrightarrow{\text{Nano Disperser}} H + OH$$
$$H + H^{\cdot} \longrightarrow H_2$$
$$OH + OH \longrightarrow H_2O_2$$

$$Fe^{2+} + 2NaOH \rightarrow Fe(OH)_2\downarrow + 2Na^{+}$$
$$3Fe(OH)_2 + H_2O_2 \rightarrow Fe_3O_4\downarrow + 4H_2O$$

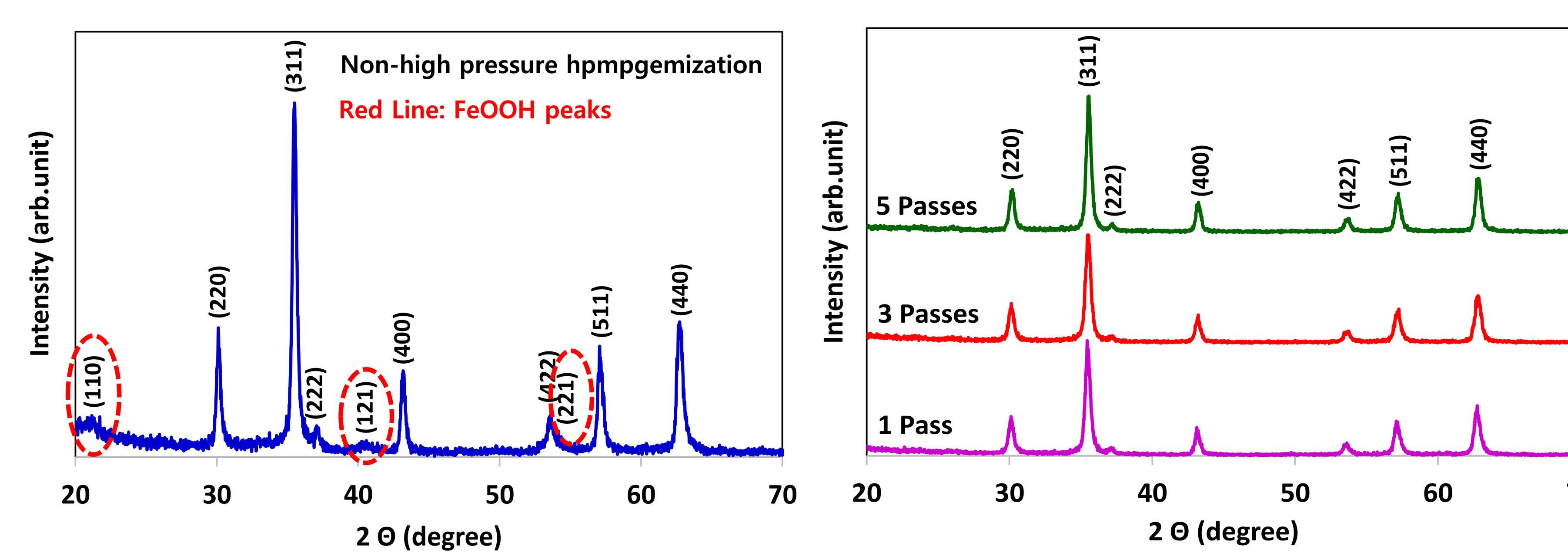


✱ Optimum conditions:
- Reaction Temp.: R.T
- Stirring Time: 5 min

High Energy (Nucleation & Crystal Growth)

- High pressure homogenization in a solution during the chemical reaction may accelerate the rate of the reaction and the crystallization may be possible at low temperature.

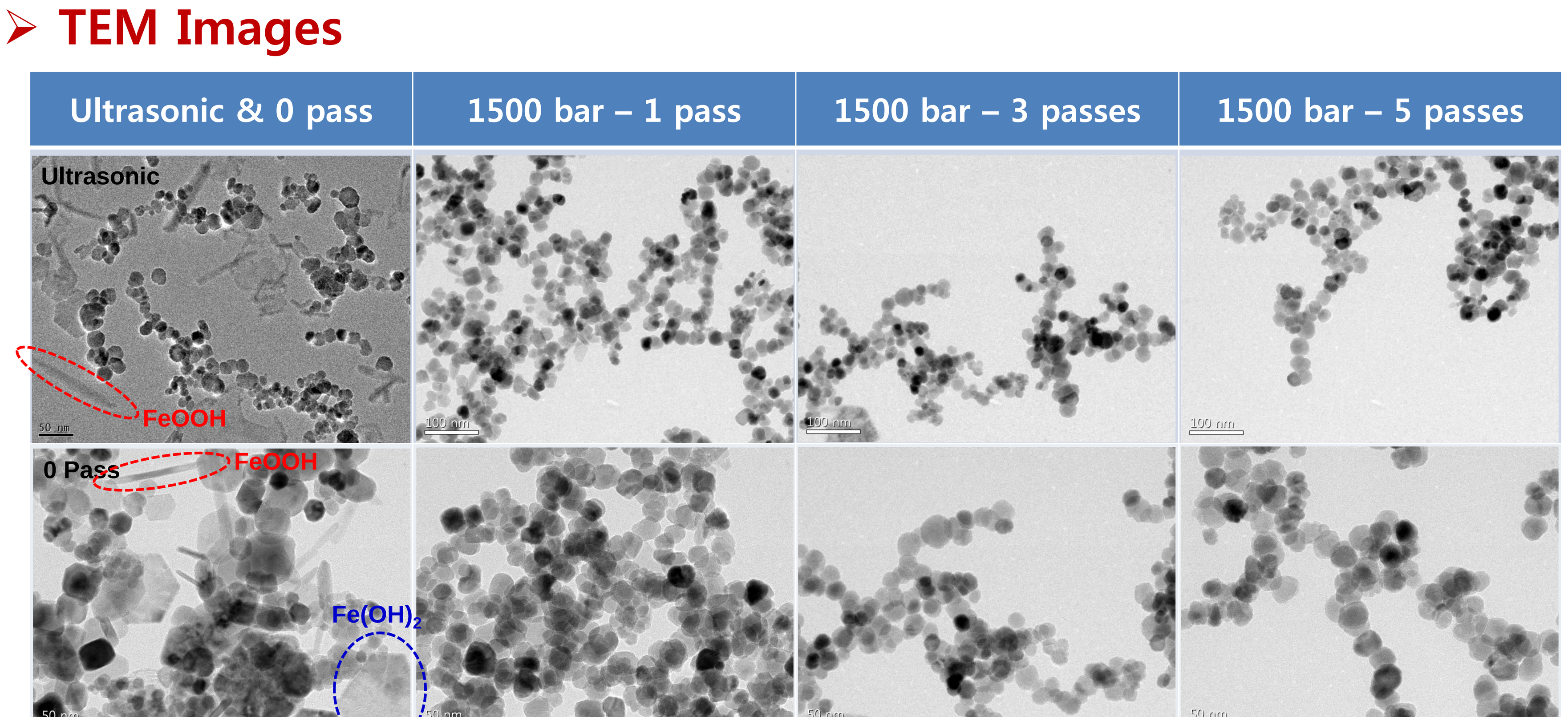
➢ XRD Patterns



- The XRD pattern of the 0 pass sample showed diffraction peaks at 2 θ = 21.1 °, 40.3 ° and 53.8 ° from the FeOOH.
- All the peaks of 1, 3 and 5 passes samples were matched to the inverse spinel Fe_3O_4

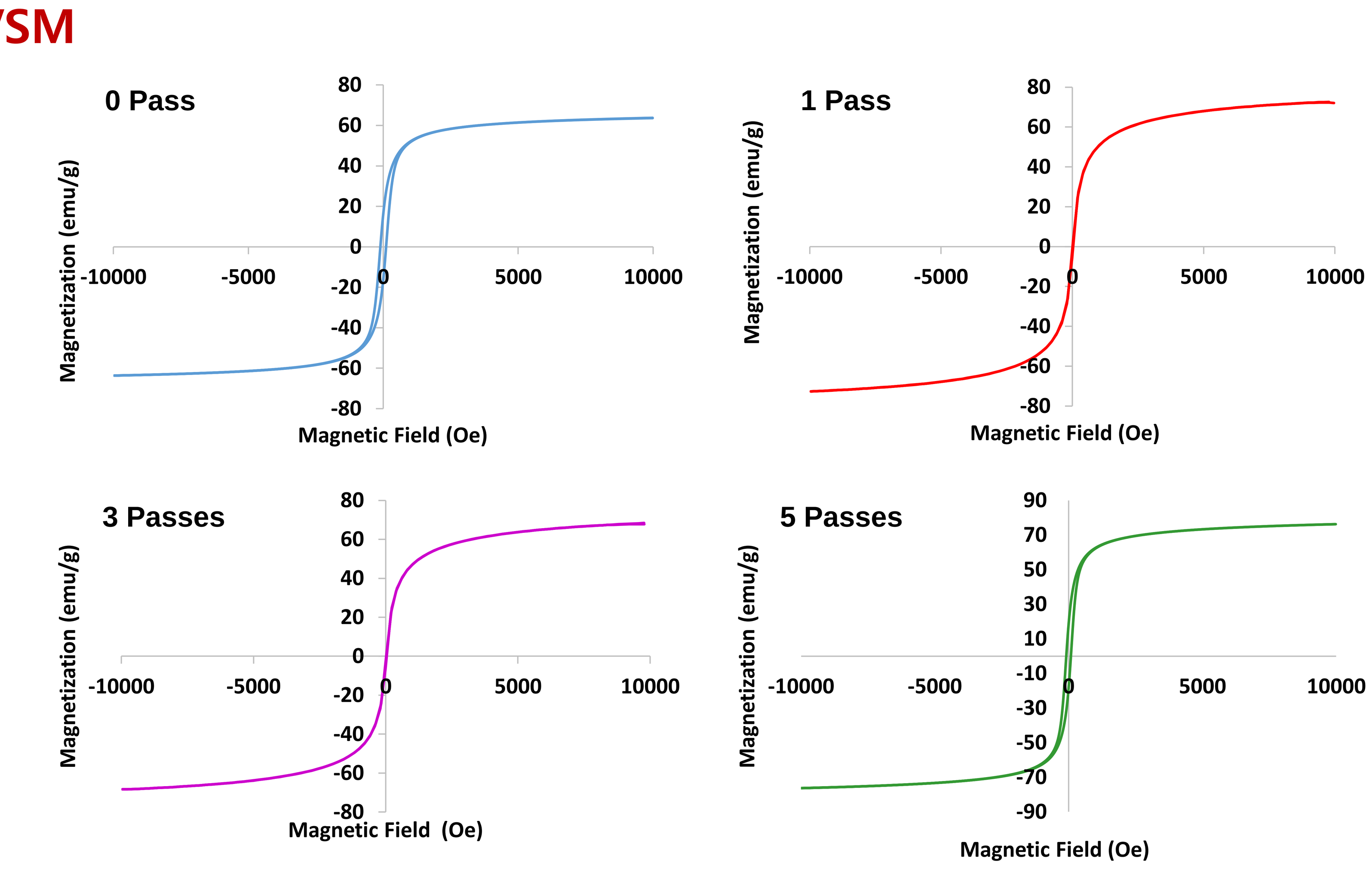
3. Results & Discussion

➢ TEM Images



- The particles synthesized through the ultrasonic method and 0 pass sample are spherical, through square and rod shapes could also be observed.
- Spherical magnetite nanoparticles were obtained for 1 to 5 passes at 1500 bar.

➢ VSM



➢ Average particle sizes and magnetic properties of the prepared magnetite nanoparticles

	Average Particle Size (nm)		Magnetic Property[b]			
	XRD[a]	TEM	Magnetization (emu/g)	Coercive (Oe)	Retentivity (emy/g)	
0 Pass	26	-	64	125	17	FeOOH $Fe(OH)_x$ Fe_3O_4
1 Pass	21	20	72	-	-	Cubic Spinel
3 Passes	19	17	67	-	-	Cubic Spinel
5 Passes	23	22	76	82	19	Cubic Spinel

[a] The particle size was calculated from the values of FWHM at the diffraction peak of 35.4 ° for Fe_3O_4.
[b] Magnetic properties were determined by VSM.

4. Conclusion

➢ We have synthesized uniform magnetite nanoparticles using the high pressure homogenizer without dispersing agent and oxidant.

➢ The X-ray diffraction patterns showed that all the samples had the inverse spinel structure of magnetite nanoparticles.

➢ The average particle size decreased with the number of passes, but after reaching 3 passes the average particle size increased.

➢ The VSM measurements revealed superparamagnetism of the nanoparticles for 1 and 3 passes at 1500 bar.

➢The uniform size, narrow distributions and superparamagnetism of these magnetite nanoparticles demonstrated their suitability for use as an MRI contrast agent, as magnetic fluids, and for targeting drug delivery systems.