THE NEOTIA UNIVERSITY

Programme: B. Sc (Hons.) Agriculture **Semester: 5th**

Course Name: Geoinformatics, Nano-technology and Precision Farming
Course Code: CCAGP 541 CCAGL 541

WORK INSTRUCTION*

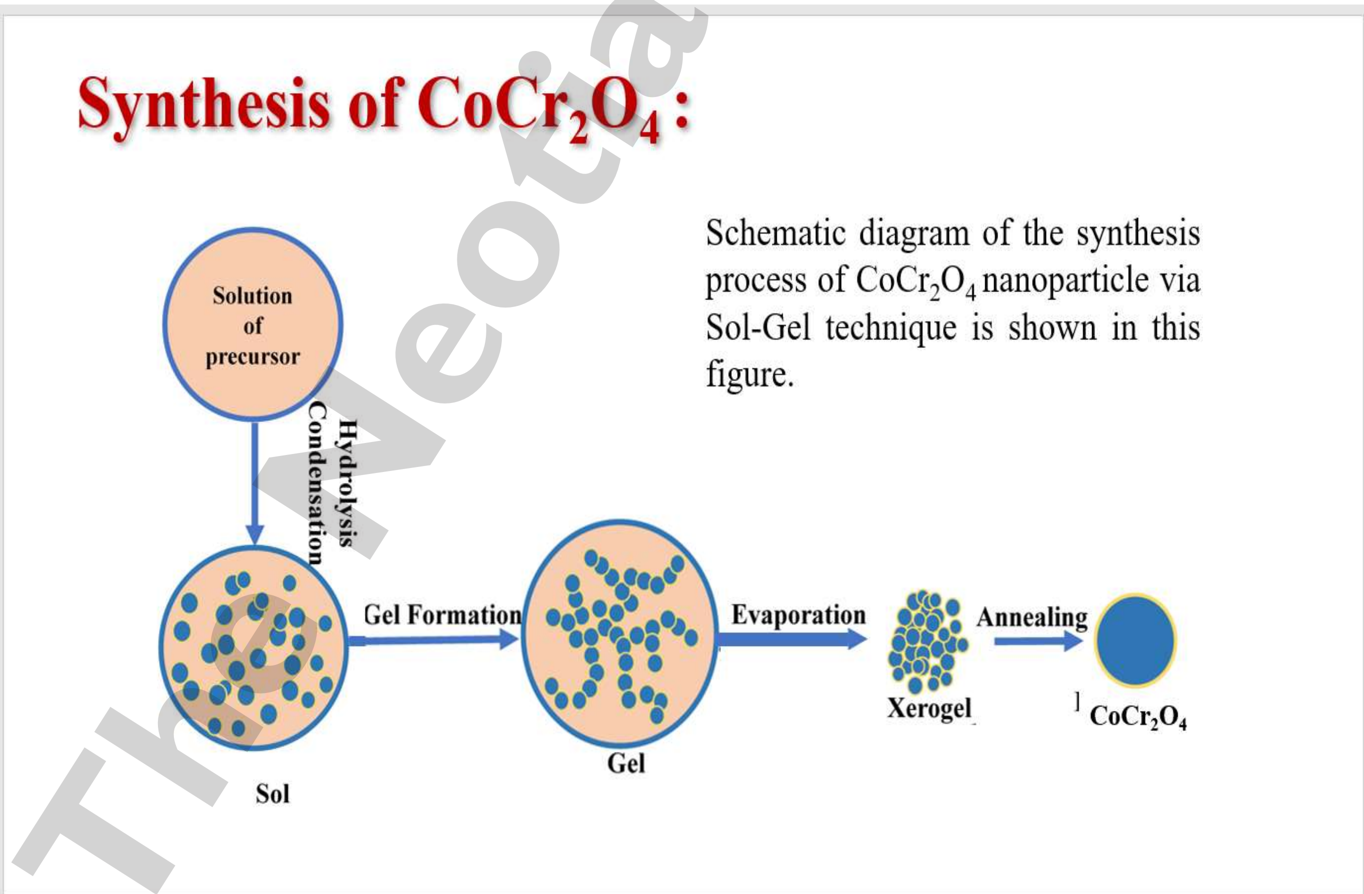
1.0 EXPERIMENT NO: 01
2.0 NAME OF EXPERIMENT: Particle size calculation from TEM/SEM image.

3.0 0BJECTIVE: Determination of average particle size of nanoparticles from transmission electron microscope (TEM)/scanning electron microscope (SEM) image

4.0 Sol- Gel technique:

Sol-gel technique is one of the bottom-up technique to synthesize nanoparticles. Figure below shows the schematic diagram of the sol-gel technique. The synthesis of cobalt chromite nanoparticle ($CoCr_2O_4$) has been demonstrated as an example with some simple steps.

1. First, we have taken 0.2 g of raw Cobalt (Co) and 0.3 g of Chromium (Cr) metal powder in a clean beaker and dissolved it into 37% hydrochloric acid. The solution is then put over a magnetic rotor for 12 hrs to form a homogenous solution.



The work instruction belongs to a part of Nano-technology of the said course

2. After that to form ionic bonding 3 g of citric acid is added in the solution and the solution is again homogenized for 12 hrs.
3. After the completion of the 2nd step the solution is put inside a vacuum oven at 50^0 C for 2 days to form a thick gel
4. The gel is dried in the vacuum oven at 60^0 C over night to form a solid cake
5. The cake was then ground in a mortar pestle and formed into a fine powder
6. After that the powder is put into an alumina boat and kept inside the chamber furnace at 600^0c for 6 hrs. to form $CoCr_2O_4$ nanoparticle.

5.0 Method to estimate particle size:

Based on the particle size data obtained from TEM or SEM figure, particle size distribution was plotted. A typical way to present the particle size and its distribution is in the form of a number-frequency histogram. A histogram is a bar graph that illustrates the frequency of occurrence of particle versus the size range. Figure 1 shows number of particles with particle size data in linear scale.

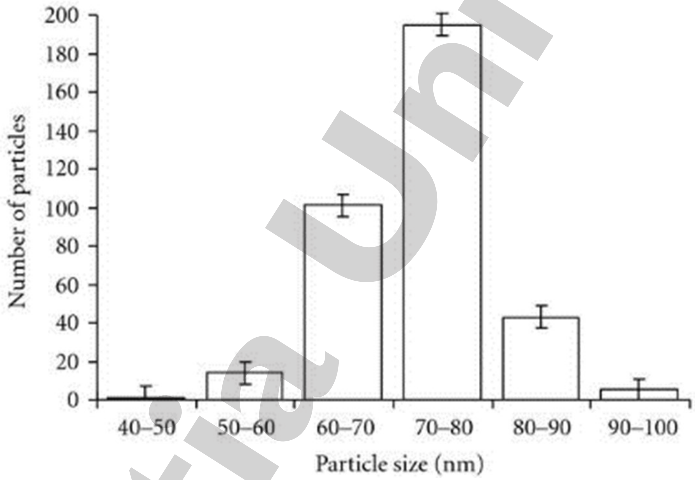
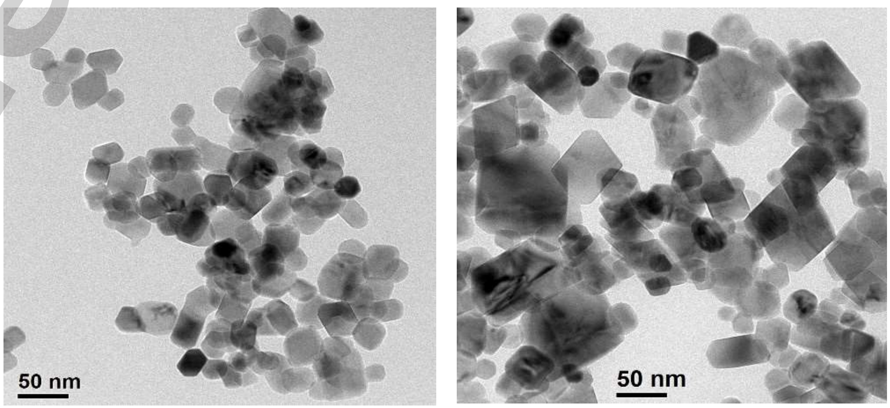


Fig. 1. Number of particles histograms showing particle size distribution of nanoparticles in linear scale.

As it is observed in Fig.1, the size distribution of the particles is skewed toward the larger end of the particle-size scale as the majority of real samples when plotted on the linear scale. The particle size range corresponding to the highest bar is estimated average particle size range of the sample.

6.0 Real Example: TEM image of $CoCr_2O_4$ nanoparticle

TEM images are used to characterize the nanoparticles. From TEM images it is possible to know about the particle size, shape, morphology, defect, structure of nanoparticles. For the calculation of average particle size, we generally use Photoshop software. Figure attached below are the TEM images of as synthesized $CoCr_2O_4$ nanoparticle.



THE NEOTIA UNIVERSITY

Fig. 2. TEM image of $CoCr_2O_4$ nanocrystalline sample

Average particle size calculation of $CoCr_2O_4$ nanocrystalline sample from TEM image:

Table-1

Determination of particle size distribution with number of particles

Sl. No.	Range of Particle Size (nm)	Average particle size (nm)	Number of particles
1	10-15	12.5	2
	15-20	17.5	13
	20-25	22.5	28
	.		
	.		
	.		
	.		
	.		
	.		
	.		
	.		
	50-55	52.5	2

How to measure the crystallite size from Graphical representation:

1. First take a mm. × mm. graph paper and choose to plot particle size along X-axis with number of particles with Y-axis.
2. Determine the appropriate coordinates of origin according to the data distribution.
3. Determine one smallest division value along both axis as per the data obtained in the table so that data distribution should cover at least 80% area of the total graph sheet.
4. Particle size region with maximum particles denote the average crystalline size.

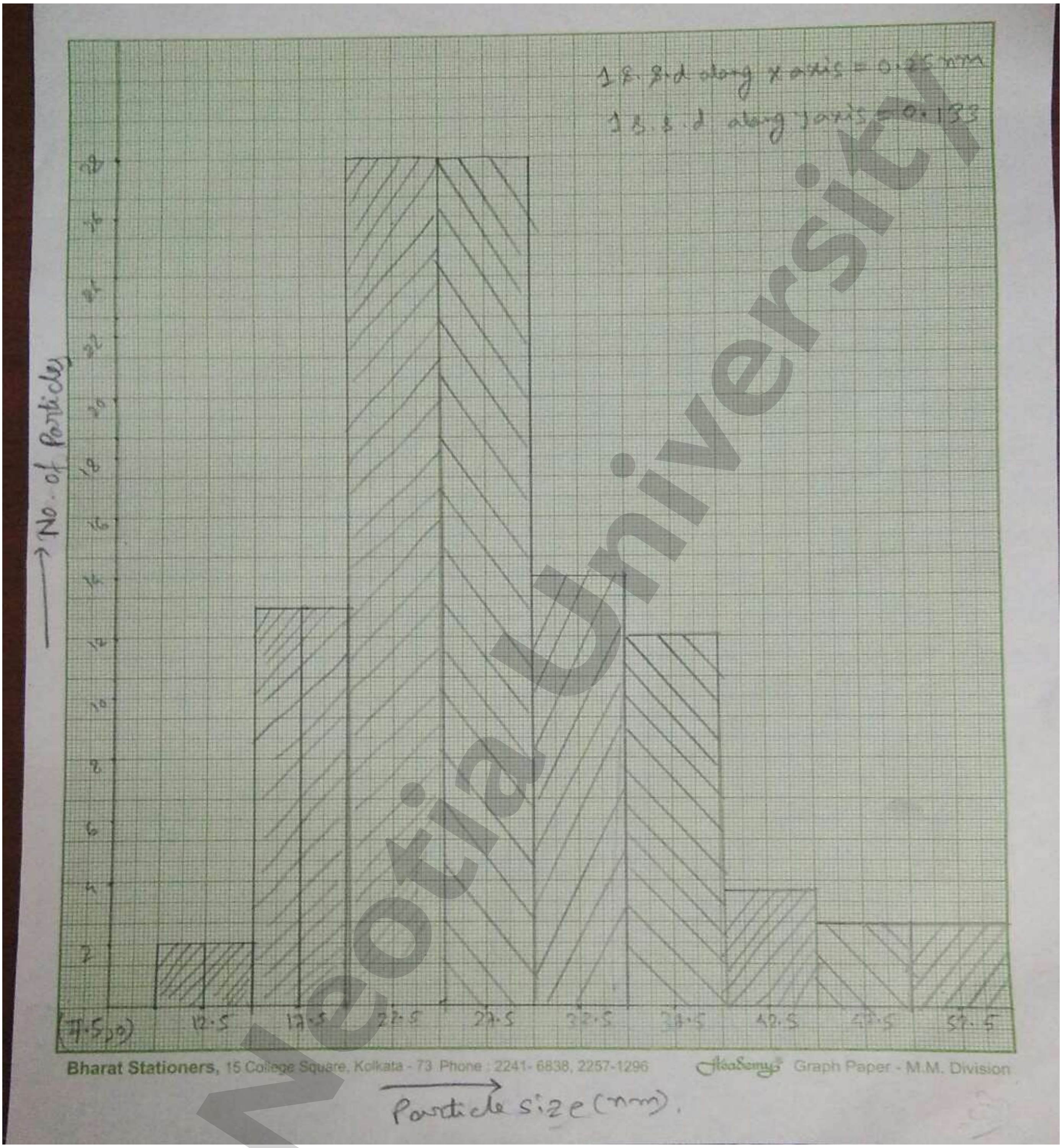
Example for method of drawing graph by referring to Table-1:

1. From table 1 we see that the diameter of particles ranges between 10 to 55 nm, which should be the range of x-axis in our graph. Since the x-axis has first average value 12.5 nm in the graph, we have chosen the origin of the x-axis as 7.5 nm. The x-axis has 190 divisions so we need to divide (55-7.5) = 47.5 nm in 190 divisions which results into 1 smallest division (S. D.) as 0.25 nm along x-axis.

2. For the y-axis, the maximum number of particles is 28 and minimum is 2. Hence, 32 particles are divided into 240 mm, which makes 1 S. D. along y-axis as 0.13333. Since the particles are not measured in fraction so there is no unit associated with it to describe the no of particles.

3. From table 1 we see that for 12.5 nm average particle size no of particle is 2. Mark the coordinate (12.5,2) in your graph paper.
4. Again, we have 2 particles in the range of 10-15 nm, to imply this information select the area between (10,2) and (15,2) and create a shaded region.

5. Similar to this method draw other regions also.



From this bar diagram we see that most of the particle resides in 20-30 nm region with maximum number of particles. Average of the region is 25 nm. Thus, from the bar diagram we can confirm that average particle size of the as synthesized $CoCr_2O_4$ nanoparticle is 25 nm.