Original Article

Synthesis and study of zinc oxide nanoparticles and their nanocomposites

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Abstract

Zinc oxide nanoparticles (ZnO NPs) was synthesized via chemical route using sodium hydroxide and zinc chloride (ZnCl2) as a precursor. Zinc oxide nanoparticles/Nanographite-oxide nanocomposite (ZnO/NGO) were synthesized by solution method. The calcination temperature of zinc oxide nanoparticles was 600 °C. The crystal structure of ZnO NPs with average size 32.99 nm was measured by XRD. FT-IR spectrum was used to confirm the presence of functional groups in ZnO and Zn(OH)2 NPs. The dynamic light scattering (DLS) gave a wider nanoparticle size distribution in two peaks with average 725 nm, also the BET and BJH analysis indicated that ZnO NPs / NGO nanocomposite have a well surface area with good nanoparticles diffusion compering as ZnO NPs. The morphology analysis by FESEM technique was given different sizes and shapes nanoparticles (oval, sheet and cubic) with average diameters extended 228.065 and 134.784 nm , 39.63 and 127 nm for Zn(OH)2 and ZnO NPs , NGO sheet and ZnO/NGO nanocomposite respectively.

Keywords: ZnO nanoparticles; Nanographite oxide, Nanocomposites

INTRODUCTION

Recent, nanotechnology has received wide and great interest from researchers, in general, it interest in the material having at least one dimension in the size range 1-100 nm.[1] For synthesis of nanoparticles, eco-friendly and safe method must be chosen., and there is a development in nanoparticles.[2] It is known that solvents and chemicals materials such as reducing factors great effect on morphology like the shape ,size and physicochemical properties of nanoparticles. [3] Generaly, there are two general approaches for the synthesis of nanomaterials"Top down" and "bottom up" for synthesis of nanomaterials, in top-to-bottom making nanoscale structures by machining, coating, atomisation, lithography and etching techniques. (physical methods and chemical methods), while in bottom-to-top ("Molecular nanotechnology") applies to building organic and inorganic structures atom-by-atom and molecule-by-molecule. (chemical methods.[4-6] The reaction conditions such as reducing agent, stabilizer factor very important for controlled of the shape and size nanoparticles.[7] Berker and his group compared between the green and chemical precipitation method to synthesis of zinc oxide nanoparticles, they obtained a different shapes and sizes of ZnO NPs, as well as they proven that the particle sizes of the prepared samples by green synthesis smaller than chemical precipitation.

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In 2014, reported when graphite was oxidized give a substance with a high water content was obtained, which allowed to obtain a colloidal substance.[9-11] To the best of our knowledge, the use of ZnO NPs and NGO sheet for ZnO/NGO composite synthesized at room temperature are gives new properties that can be used in several chemical applications. Hence the present study was carried out to synthesize and characterize the ZnO nanoparticles and ZnO/NGO nanocomposite using simple chemical method and study their properties.

Experimental Part

Materials

Zinc chloride (ZnCl2), sodium hydroxide (NaOH), ethanol, conc. H2SO4, conc. HNO3, distilled water and nanographite were obtained from Department of Chemistry, College of Education for Pure Science, University of Diyala, Iraq.

Instruments and Apparatus

The samples analysis by XRD, FESEM, DLS and BET Techniques of the prepared compounds and the study of their properties were carried out in the College of Science/University Tehran-Iran, while FTIR spectrum for ZnO NPs are obtained in Diyala University/College of Education for Pure Sciences.

Preparation of Zinc oxide nanoparticles

For the purpose of obtaining zinc oxide nanoparticles, dissolve (0.5 g, 0.00366 mole) of zinc chloride (ZnCl2) in (500 mL) distilled water, add to the zinc chloride solution with constant stirring, drops of sodium hydroxide solution (NaOH, 1M, until we get PH= 14) after few minuts a white precipitate of zinc hydroxide nanoparticles (Zn(OH)2 NPs) was formed. The zinc hydroxide nanoparticles collected and wash several times with distilled water until we reach pH = 7, then dried the precipitate at (80 ° C, 16 hours). After that the Zn(OH)2 NPs calcinated at (600 ° C , 6 hours) for obtained ZnO nanoparticles.

Synthesis of ZnO/NGO composite

To obtain oxidized nanographite (NGO), a mixture of nitric and sulfuric acid (20 mL, 3:1) was added to nanographite (0.5g) in ultrasonic at (80°C , 4 hours). Using solution method the ZnO/NGO composites were synthesized (0.1g NGO sheet : 0.3g ZnO NPs) . An alcohol solution was prepared by suspended (0.1g) NGO and (0.3g) ZnO NPs in ethanol (20 mL) at room temperature with constant stirring; the mixture was placed in ultrasonic apparatus at 3 hour. The nanoparticles are completely suspended with NGO sheet, the solvent was evaporated to obtain a ZnO/NGO as a binary composite. Figure 1.

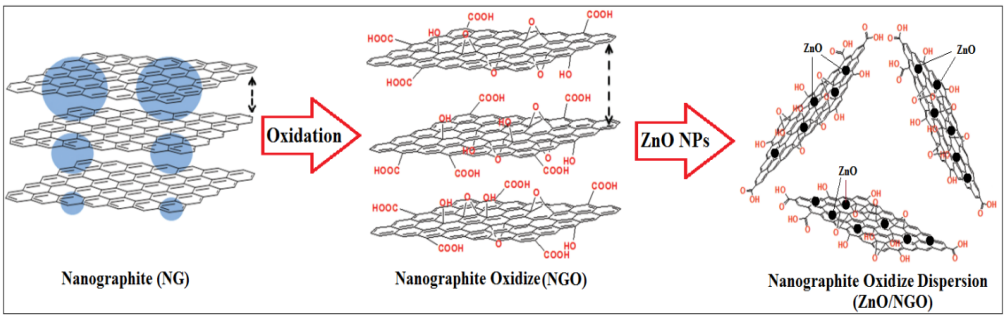

Fig.1. scheme of ZnO/NGO composite formation Zn(OH)2.

Result And Discussion

FTIR analysis

FT-IR spectra of Zn(OH)2 and ZnO nanoparticle showed in Figure 2, two frequencies at 3379 cm−1 and 3417 cm−1 return to bond O-H stretching vibration in the Zn(OH)2 nanoparticles, while a bands at 462-555 cm-1 refer to Zn-O stretching bond. The presence of ZnO NPs was identified by the presence of absorption band approximately at 3443 cm−1 for the bulk zinc oxide (ZnO) represented to the H2O in the air and ZnO sample are responsible for existence of such peaks (Majd Abusalem et al,2019), and it is Zn-O stretching bond at 493 cm-1 which confirmed the FTIR spectrum presence that ZnO nanoparticles compared to

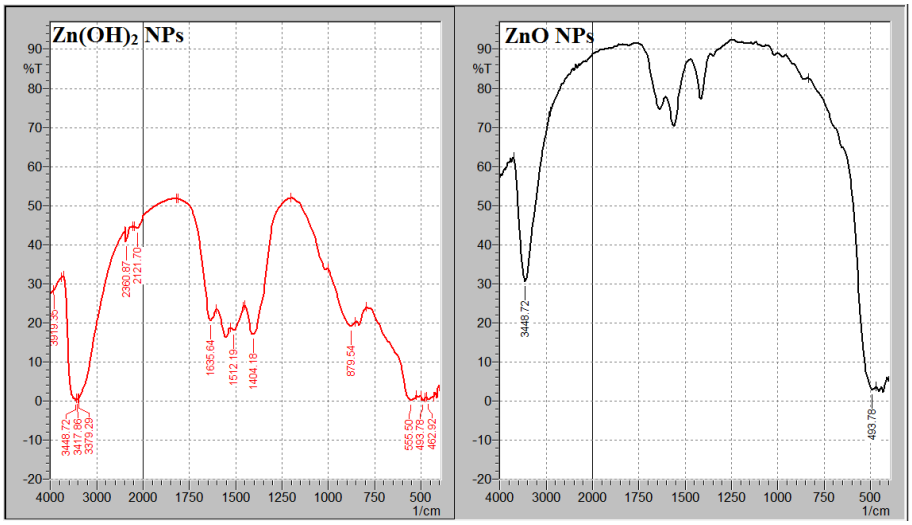

Fig.2. FTIR spectrum for Zn(OH)2 and ZnO NPs

Figure 3, because of the chemical inertness of nanographite (NG) sheet, not shown any important peak in the FTIR spectra of nanographite , while, showed the peak at 3433 cm-1 represent (–OH) stretching vibration, with two peak at 2862 and 2954 refer to symmetric and asymmetric stretching vibration of (–CH2–). The sharp peak at 1720 and 1581 cm-1 due to the carbonyl stretching (C=O) bond with stretching bending vibration of C=C groups in the NGO sheet respectively. The stretching vibration of C-O bond in carboxylic acid and C-OH of alcohol are revealed by the weak peaks at 717cm-1,1242 cm-1 and 1080cm-1 respectively.

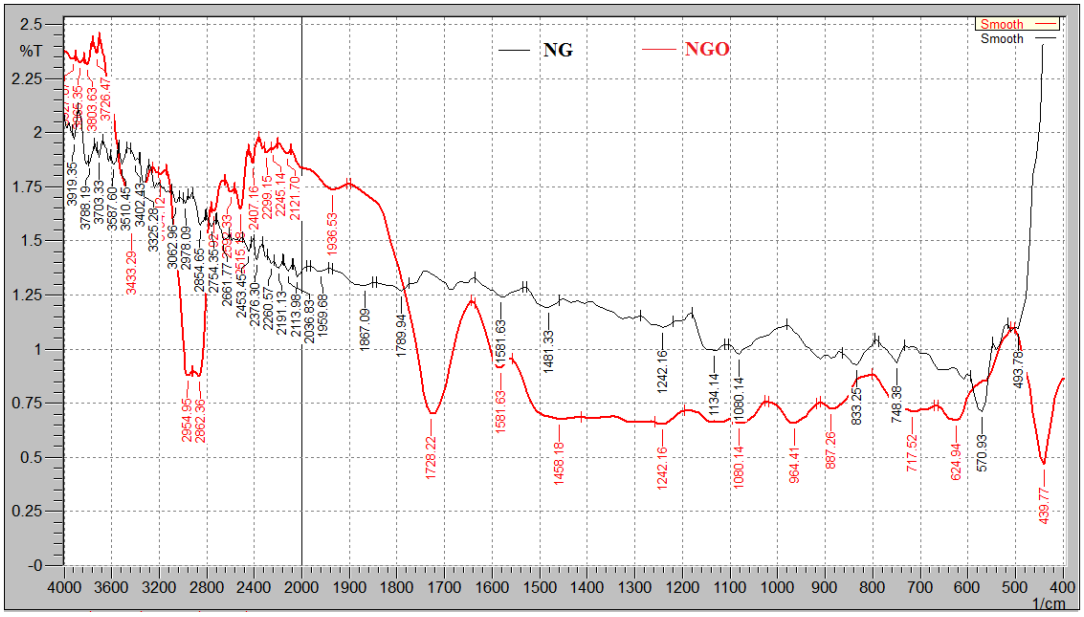

Fig.3. FTIR spectra of NG and NGO

FTIR of ZnO/NGO nanocomposite shown in the figure 4. In the case of ZnO/NGO nanocomposite, there is a changing in FTIR spectrum of NGO which lead to lower the oxygen functional groups in the nanocomposite. the peak at 1620 cm-1 and 450 cm-1refer to nanographite sheets (C=C) and stretching vibration of ZnO, hence و this indicates the dispersion of ZnO on NGO matrix. The O-H stretch was observed at 3400 cm-1in the ZnO/NGO structure.

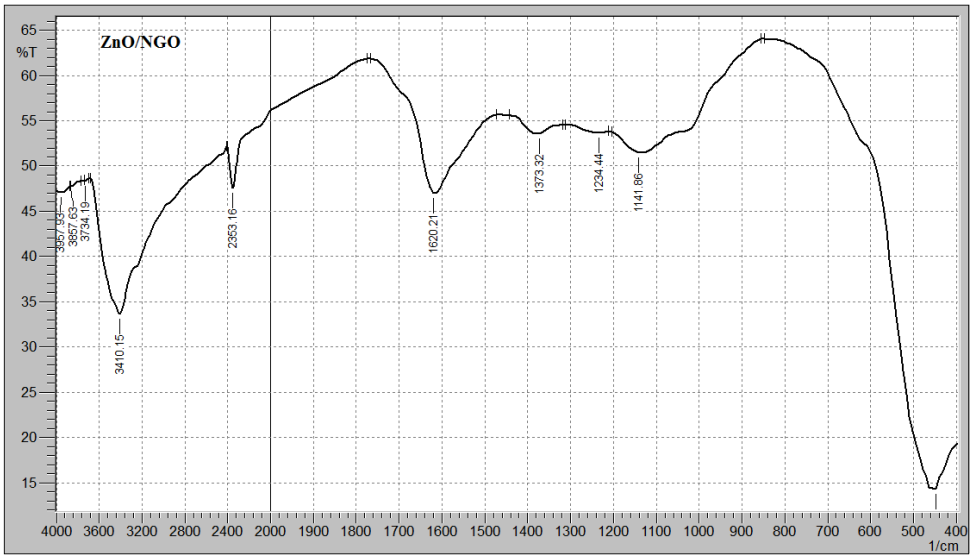

Fig.4. FTIR spectrum for ZnO/NGO nanocomposite

XRD analysis

Figure 5 shows that the 2 theta at 31.95, 34.61, 36.43, 47.72, 56.76 ,63.02, 66.53, 68.10 and 69.23 prove of ZnO nanoparticles with the average crystallite size at 32.99 nm.

2 Theta (degree)	FWHM	D, nm	D, nm Average
31.9565	0.246	35.1	32.99091
34.6123	0.246	35.34	
36.4366	0.246	35.52	
47.7225	0.2952	30.75	
56.7637	0.2952	31.96	
63.0255	0.3444	28.27	
66.5311	0.246	40.36	
68.101	0.3444	29.09	
69.2331	0.3936	25.63	
72.7131	0.2952	34.92	
77.0962	0.2952	35.96	

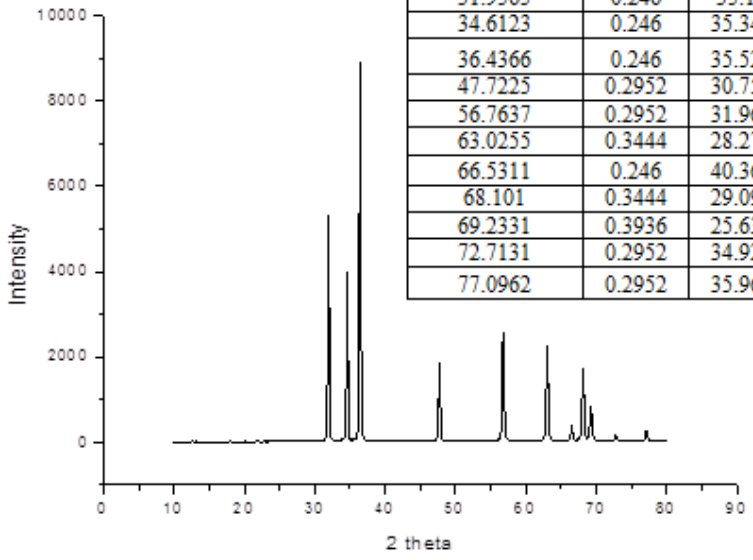

Fig 5 .XRD pattern ZnO NPs chemical method

FESEM

FESEM images of the Zn(OH)2 NPs, ZnO NPs ,NGO sheet and ZnO/NGO nanocomposite are shown in Figures (6-9) revealed oval ,sheet and cubic nanoparticles respectively. The oval nanoparticles belong to the Zn(OH)2 and ZnO NPs prepared by the chemical precipitation method with average diameters 134.784 nm and 228.065 nm respectively. Sheet shapes with average diameters 39.63 nm refer to NGO, while average diameters about 127 nm with cubic shape refer to ZnO/NGO nanocomposite.

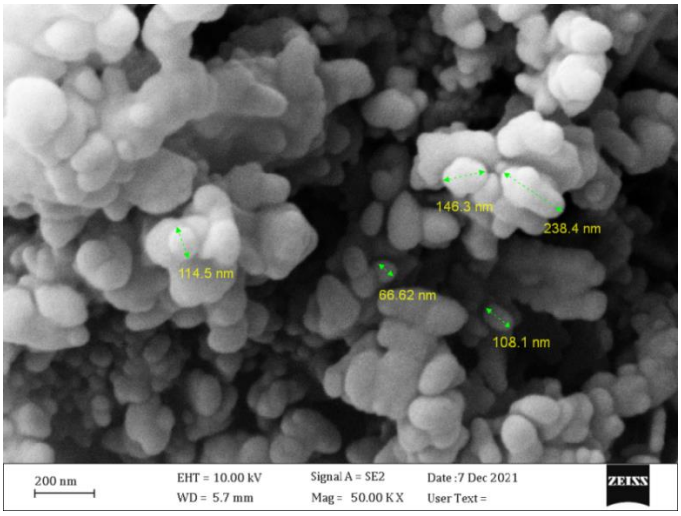

Fig 6. FESEM image of Zn(OH)2 nanoparticles

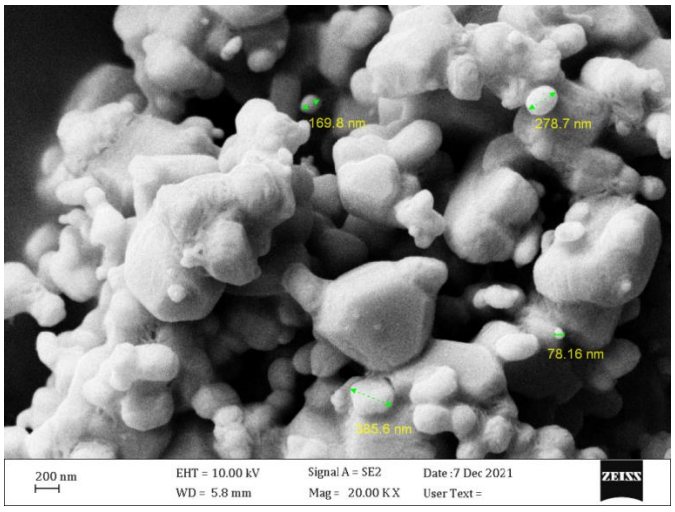

Fig 7. FESEM image of ZnO nanoparticles

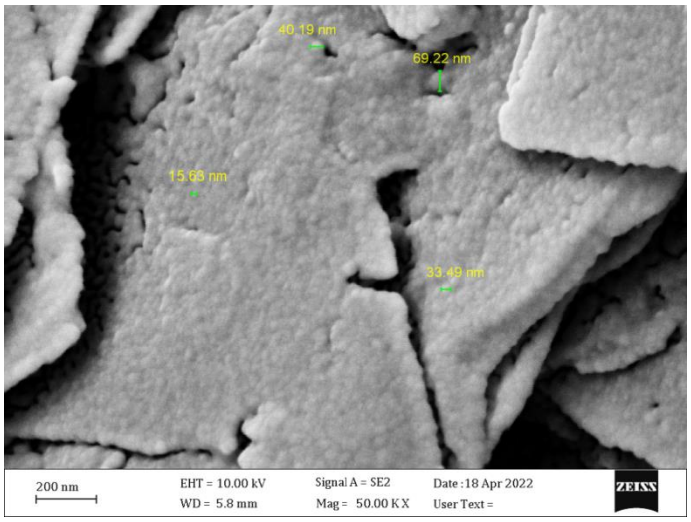

Fig 8. FESEM image of NGO

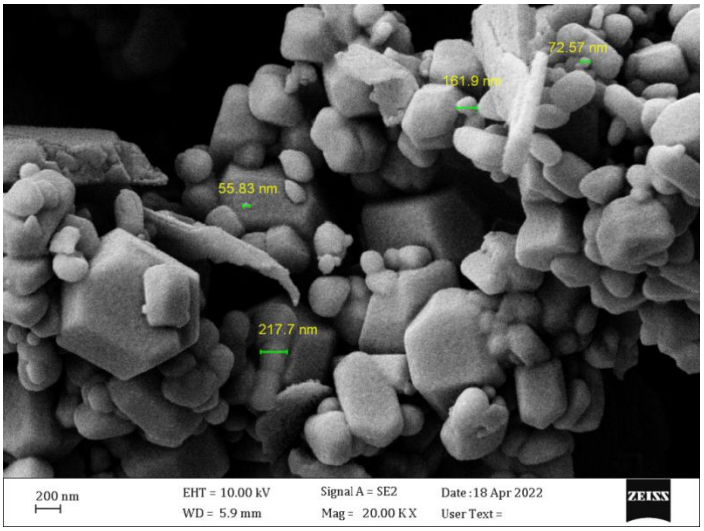

Fig 9. FESEM image of ZnO /NGO composite

DLS analysis

By DLS technique were obtained the distribution size of ZnO nanoparticles at range approximately from 200 nm to 1500 nm in the form of two peaks Figure 10.

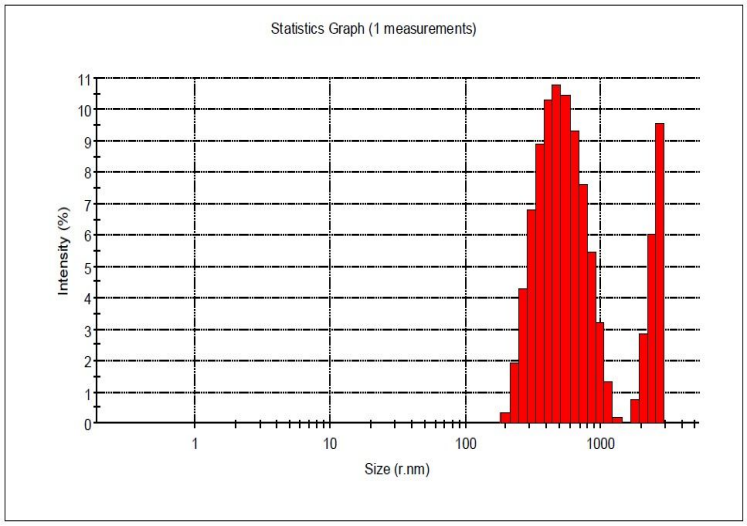

Figure 10. Distribution of ZnO nanoparticles according to intensity

BET analysis

By the Brunauer Emmett-Teller (BET) equation were specific surface areas obtained fit for N2 gas adsorption isotherms of the zinc oxide nanoparticles and their composites samples, while from BJH analysis, the diameter and volume pores were obtained. The BET results shown in the table and figures 11 - 13.

Sample	BET specific surface area (m2 g-1)	Lag. specific surface area (m2 g-1)	Pore Volume (cm3 g-1)	Pore Diameter (nm)
ZnO	1.2589	1.6845	0.0043254	13.743
NGO	3.134	7.843	0.006961	9.2471
ZnO/NGO	42.821	59.739	0.08921	21.375

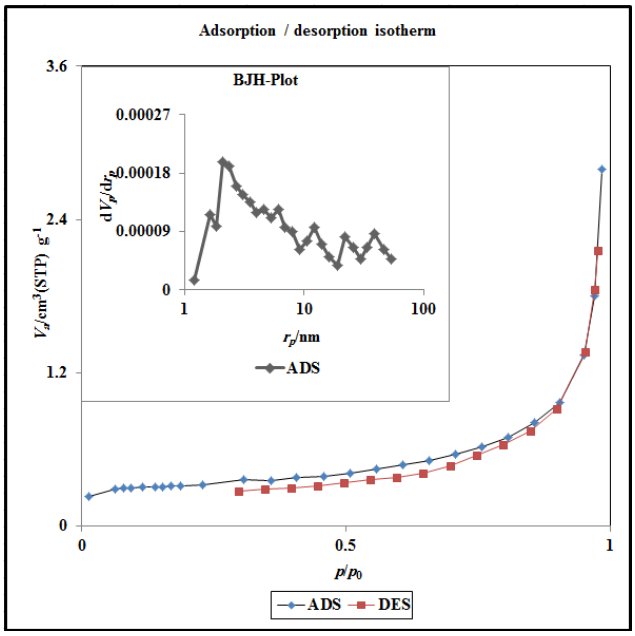

Fig 11. BET and BJH (insert) plots of ZnO nanoparticles

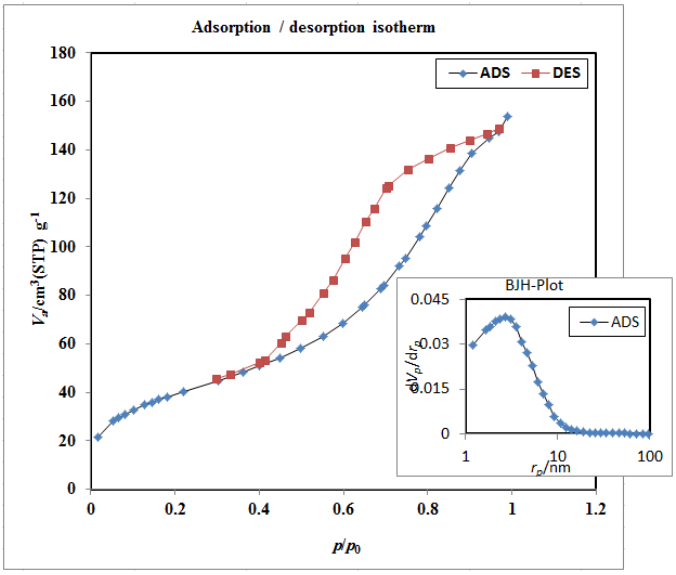

Fig 12. BET and BJH (insert) plots of NG sheet

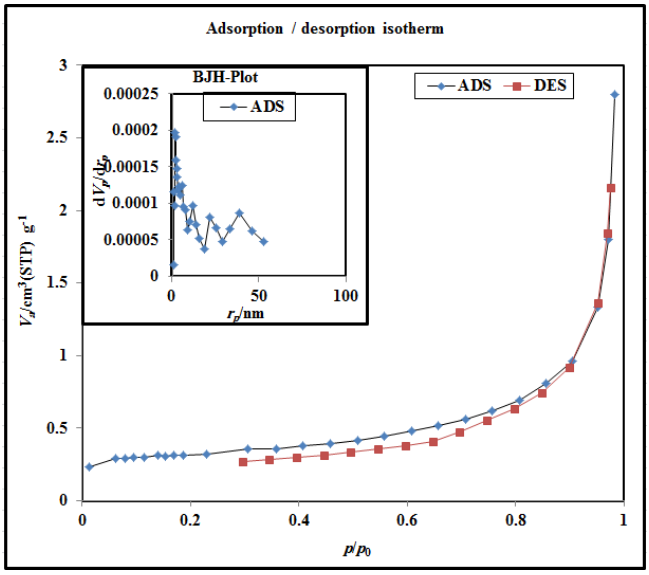

Fig 13. BET and BJH (insert) plots of ZnO/NGO nanocompesite

Conclusions

ZnO nanoparticles was prepared from zinc chloride by chemical precipatation was Successful. ZnO/NGO nanocomposite was prepared from ZnO nanoparticles and Nanographite by chemical solution method was Successful too. They were studied by FESEM, XRD, DLS and BET to get the shape, size distribution and surface area of them. Examination of ZnO nanoparticles and ZnO/NGO composite were indicated that ZnO/NGO nancomposite have a high surface area, while NGO sheet showed smaller size of nanoparticles. Both ZnO nanoparticles and ZnO/NGO nanocomposite were has cubic shapes with average size about 134.784 nm and 127 nm respectively.

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