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Synthesis and Thermal treatment of Zn-Cr and Ni-AL Nanoparticles: Structural and Morphological Characterization

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Abstract:

Layered double hydroxides of Zn-Cr and Ni-AL were synthesized by co-precipitation technique. thermal treatment product(MMO) was made at (600)°C. In (XRD) results all samples Polycrystalline. The results of (FE-SEM) show that subsequent heat treatments of Zn-Cr LDHs and Ni-AL LDHs at 600 °C lead to an increase of the nanoparticles size to 51.53 nm and 51.17 nm, respectively. AFM images show a good distribution for nanoparticles on the surface and, the particle size increases after heat treatment at 600 °C. low cost as well as simple preparation for prepared Layered double hydroxides offers an ideal way for obtain nanoparticles with small crystallite sizes, the high crystallinity and large surface area.

Keywords: Nanoparticles; Layered double hydroxides; Mixed metal oxide; Co-precipitation; Thermal treatment.

Note: The research is based on a PhD dissertation.

Introduction:

Nanotechnology is a known as the field research from the last century. For this technology various revolutionary developments since Richard P.Feynman well lecture e “There’s Plenty of Room at the Bottom” in1959[1]. Nanotechnology has had an impact on both society and the economy. By nanotechnology, most current environmental, medical, and industrial issues have been remedy[2]. Nanotechnology field contain various types materials at nanoscale level, nanoparticles are wide category from this materials which have one dimension at least (> 100 nm)[3]. Depending on nanoparticles’ morphology, size and chemical properties, It divided into Metal NPs, Semiconductor NPs, Polymeric NPs and Carbon-based NPs[4]. The bottom-up approach involves several chemical techniques for producing

metal nanoparticles, like chemical reduction, Photochemical, Sol-gel, and co-precipitation. Within the co-precipitation technique, metal salts are combined in the aqueous solution and the ions of those metals are precipitated by adjusting the pH and adding alkaline of ammonia, sodium hydroxide, or ammonium hydroxide. Following the deposition procedure, the precipitated substance is rinsed, then dehydrated and combusted. The resulting specks are nano-scale, comparable in size, and exceptionally effective. This is one of the benefits of the co-precipitation approach besides being simple and affordable. By using the co-precipitation technique in preparation, numerous elements can be managed and altered, like concentration and temperature, which are connected to dissolution[5][6]. LDHs have positively charged layered structure which motivates characteristics such as anion exchangeability, mobility, and surface basicity. The anions present between LDHs layers and water are unstable, and consequently, exchange reactions can be utilized to replace interlayer anions for several inorganic or organic anions. The ion-exchange process involves the interlayer anions reciprocity with other guest anions introduced into the structure of LDHs[7].The LDHs capacity for exchange the interlayer anions distinguishes LDHs as carriers toxic anions or scavengers for it[8-11] .the LDHs formula represented by$[M^{2+}_{1-x}M^{3+}_{x}(OH)_2](A^{n-})_{x/n}\cdot mH_2O$ .M^{2+} a divalent metal cation (Mg^{2+}, Zn^{2+}, Ca^{2+},...), M^{3+} a trivalent cation (Al^{3+}, Fe^{3+}, Cr^{3+}, ...) and the exchangeable anion (CO_3^{2-}, NO_3^{2-}, SO_4^{2},…)[12]. LDHs can be prepared using various different methods such as co-precipitation, urea decomposition-homogenous precipitation, reconstruction and hydrothermal methods[13]. LDHs calcinated results mixed metal oxides which are in the homogenous mixture form have small crystallite sizes, surface basicity and large surface area. Both LDHs and MMO have a high catalytic activity[7]. LDHs and LDH-based composites have wide and various applications such as materials bio-related, electrochemistry, photofunctional and catalysts due to their characterized included the structure layered, confined microenvironment and tunable composition[14] and water treatment [15]. Metal nanoparticles, metal oxide ،polyoxometalates and carbon-based nanomaterials, they all represent various modication strategies for LDHs[16-19].

2. Experimental Part

The LDH compound was synthesized Based on co-precipitation method, one of the preferred chemical methods for preparing nanoparticles. Nickel nitrate hexahydrate ($Ni(NO_3)_2 \cdot 6\ H_2O$) was thoroughly mixed along with aluminium nitrate nonahydrate ($Al(NO_3)_3.9H_2O$) for 30 minutes considering a molar ratio of 4:1, respectively, in deionized water 150 mL; a stable pH level of the attained solution (pH = 9± 0.1) was recorded as a function of molar sodium hydroxide (NaOH) 2M that added drop by drop during the reaction. addition throughout the reaction under constant stirring rate of 750 rpm. The aging process done By stirring for a full hour to resultant mixture . Subsequently, the greenish slurry was filtered then using deionized water for washed several times .For 12 hours at 65 °C in air oven, The attained precipitate was dried later. After grinding the product, we obtained Ni-AL LDH in a forma fine powder that stored for later use. With thermal treatment temperatures (600 °C), fine powder was subjected in constant time, 2 hour and heating/cooling rate10 °C/minute to produced MMO. The samples are particular as LDH and MMO-T, T Which represents thermal treatment temperature.

By following the previous steps and mixing zinc nitrate hexahydrate ($Zn(NO_3)_2. 6H_2O$) with chromium nitrate nonahydrate ($Cr(NO_3)_3. 6H_2O$) In particular a molar ratio of 4:1and at (pH = 8 ± 0.1) To be the output the purple slurry, we prepared Zn-Cr LDH fine powder After completing the washing, drying and aging steps approved above .The product was treated with the same thermal treatment temperature and formed MMO. In many Methods, the prepared samples were characterized, including: X-ray diffraction technique for structural analysis (XRD, Shimadzu-XRD-6000), the XRD was performed ,λ = (0.15417) nm radiation, (30) mA, and (40) kV, while morphological investigation via atomic force microscopy(FlexAFM,NanosurfAG,2010)and field emission scanning electron microscopy (FE-SEM, Mira3-XMU, TESCAN, Japan,).

3. Results and discussion

Figure 1 (a) expound the XRD patterns of Zn-Cr LDHs, a dual-phase nanostructured system clearly demonstrate for CrO_2 NPs at (110), (101), (111), (211) planes, and (310) and ZnO NPs at (100), (002), (101), (102), (110), (103), and (112) planes confirming the high crystallinity for hexagonal tetragonal CrO_2 and ZnO phases[20]. Figure 1 (b) elucidate the

XRD patterns of the thermally treated produced (MMO-600) for Zn-Cr LDHs. the (100), (002), (101), (102), (110), (103), (112), and (201) planes assigned to the hexagonal wurtzite of ZnO, while the (110), (111), (211), and (221) planes for tetragonal CrO_2 .The sharp peaks confirmed a high degree of crystallinity

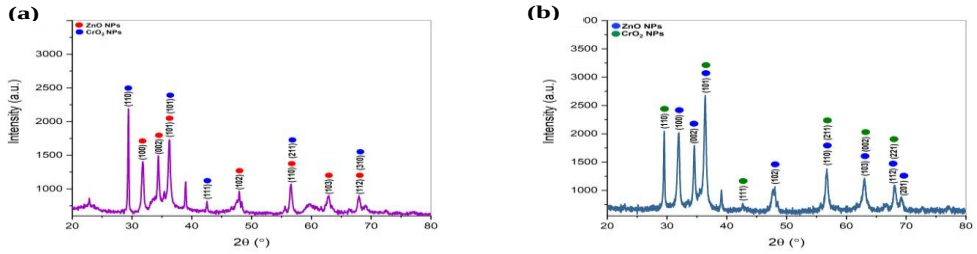

Fig. (1) XRD patterns of the prepared (a) Zn-Cr-LDH, (b) Zn-Cr-MMO-600.

Table (1): The structural properties of Zn-Cr-LDH and Zn-Cr--MMO-600.

	2 θ (deg)	FWHM (deg)	2 θ (Rad)	FWHM (Rad)	D (nm)	Matched by
Zn-Cr-LDH	29.374	0.378	0.256	0.007	21.726	01-079-2205 01-076-1232
	31.793	0.378	0.277	0.007	21.852	
	34.398	0.378	0.300	0.007	22.000	
	36.253	0.567	0.316	0.010	14.743	
	42.530	0.567	0.371	0.010	15.035	
	47.863	0.567	0.418	0.010	15.329	
	56.564	0.378	0.494	0.007	23.865	
	62.877	0.378	0.549	0.007	24.633	
	67.953	0.378	0.593	0.007	25.343	
MMO-600	29.495	0.566784	0.257	0.010	14.488	01-079-0205 01-075-0078
	31.941	0.377856	0.279	0.007	21.860	
	34.556	0.377856	0.302	0.007	22.010	
	36.383	0.377856	0.318	0.007	22.122	
	42.696	0.566784	0.373	0.010	15.043	
	47.812	0.755712	0.417	0.013	11.494	
	56.725	0.377856	0.495	0.007	23.883	
	63.018	0.377856	0.550	0.007	24.651	
	66.706	0.566784	0.582	0.010	16.774	
	68.084	0.47232	0.594	0.008	20.290	
	69.202	0.47232	0.604	0.008	20.426	

Figure 2 (a) demonstrates the XRD patterns of Ni-AL LDHs. LDH patterns to indicate interlayer structure with 2θ values which are related(012), (015), and (009) to the **NiO_2** nanoparticles phase while (104), (024), (116), (214), and (125) planes for Al_2O_3 nanoparticles. The high crystallinity demonstrated by The sharp and defined peaks[21].Figure 2 (b) demonstrates the XRD patterns of the thermally treated producted (MMO-600) for Ni-AL LDHs. In particular, Thermal treatment of 600 °C resulted in highly formed metal oxide after thermal treatment. the **NiO_2 NPs** diffraction peaks at (012), (015), and (018), whereas(111), (022), (113), (131), (202), (104), (204), (134), (151) planes correspond to Al_2O_3 NPs. The pattern showed the successful synthesis of Zn-Cr-MMO at nanostructured and crystallinity in a high degree [22].

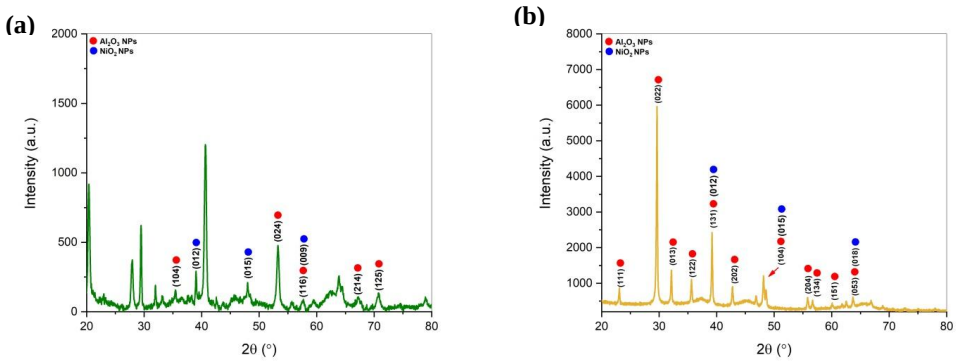

Fig. (2) XRD patterns of the prepared(a) Ni-AL -LDH, (b) Ni-AL -MMO-600.

Table (2): The structural properties of Ni-AL-LDH and Ni-AL-MMO-600.

	2 θ (deg)	FWHM (deg)	2 θ (Rad)	FWHM (Rad)	D (nm)	Matched by
Ni-AL-LDH	35.380	0.567	0.309	0.010	14.706	01-075-0785 01-085-1977
	39.106	0.567	0.341	0.010	14.868	
	48.125	0.756	0.420	0.013	11.508	
	53.277	0.378	0.465	0.007	23.512	
	57.757	0.567	0.504	0.010	16.001	
	67.331	0.945	0.588	0.016	10.101	
	70.767	0.567	0.618	0.010	17.185	

MMO-600	23.048	0.377856	0.201	0.007	21.449	01-088-0107 01-085-1977
	29.594	0.566784	0.258	0.010	14.492	
	32.106	0.566784	0.280	0.010	14.579	
	35.612	0.566784	0.311	0.010	14.716	
	39.187	0.377856	0.342	0.007	22.308	
	42.744	0.566784	0.373	0.010	15.046	
	48.303	0.566784	0.422	0.010	15.355	
	55.778	0.566784	0.487	0.010	15.852	
	56.777	0.377856	0.495	0.007	23.889	
	60.104	0.377856	0.525	0.007	24.280	
	63.731	0.377856	0.556	0.007	24.746	

The FE-SEM results showed the topographies of the samples are illustrated in Figure3(a,b,c,d). In general higher nanoparticle distribution density and aggregation after the thermally treated and lead to an increase of the size of the nanoparticles for both compounds which could possibly revealed to porous nature. Zn-Cr LDHs nanoparticles size increase from 33.55nm to51.53nm (Figure3(a,b)),while Ni-AL LDHs nanoparticles size from31.52nm to 51,17nm (Figure3 (c,d)) which are agreement with the results for [23][24].

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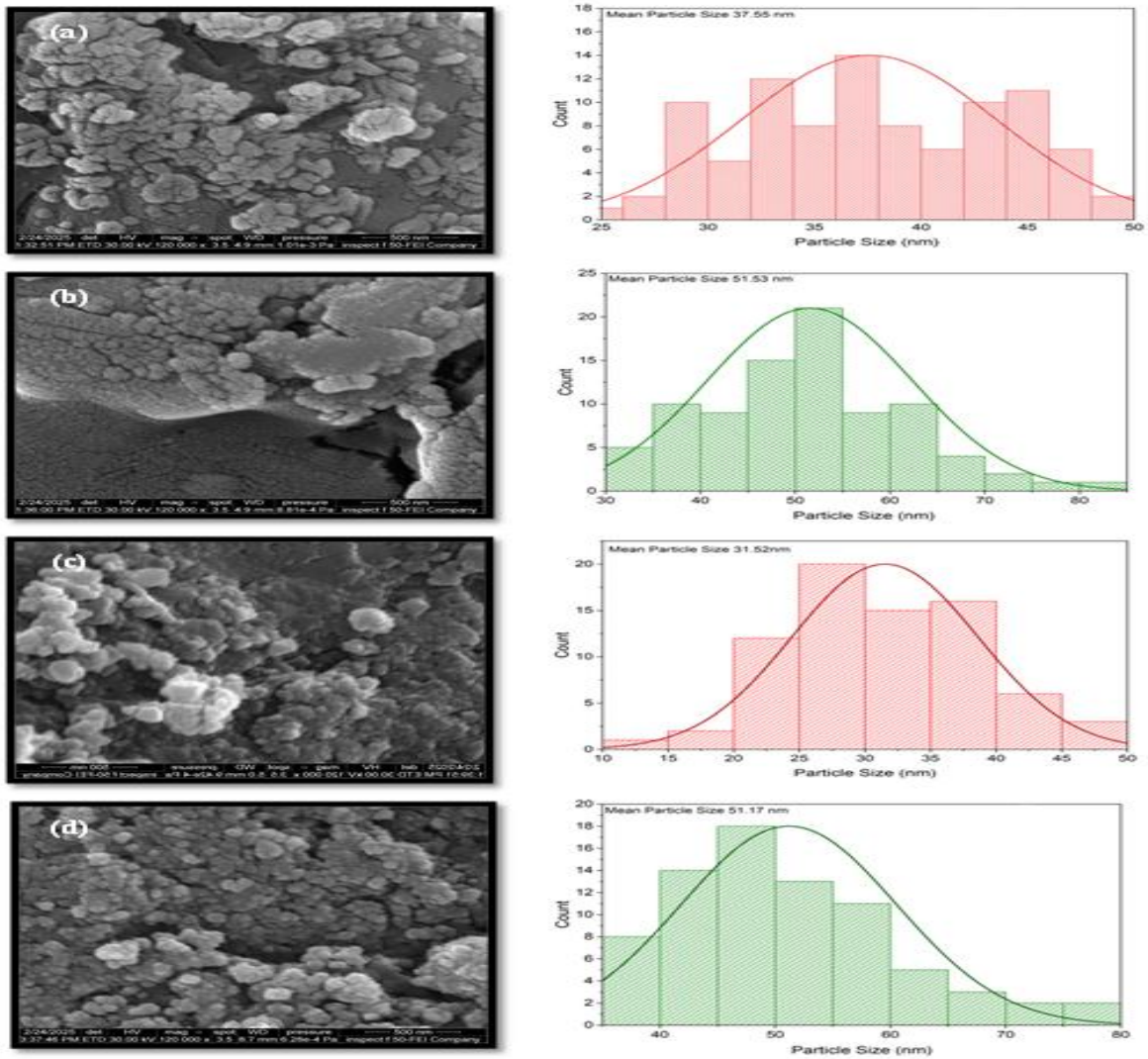

Fig. (3) FE-SEM of the prepared (a) Zn-Cr-LDH, (b) Zn-Cr-MMO-600, (c) Ni-AL-LDH, (d) Ni-AL-MMO-600.

AFM measurements included imaging in two and three dimensions (2D and 3D) as showing Figure4 (a,b,c,d) where it was calculated the grain size values, square root rate and surface roughness as explained in the table (1). AFM measurements confirmed that all samples have a nanostructure and after heat treatment at 600 °C, the particle size increases which agree with the FE-SEM results. AFM images show a good distribution for nanoparticles.

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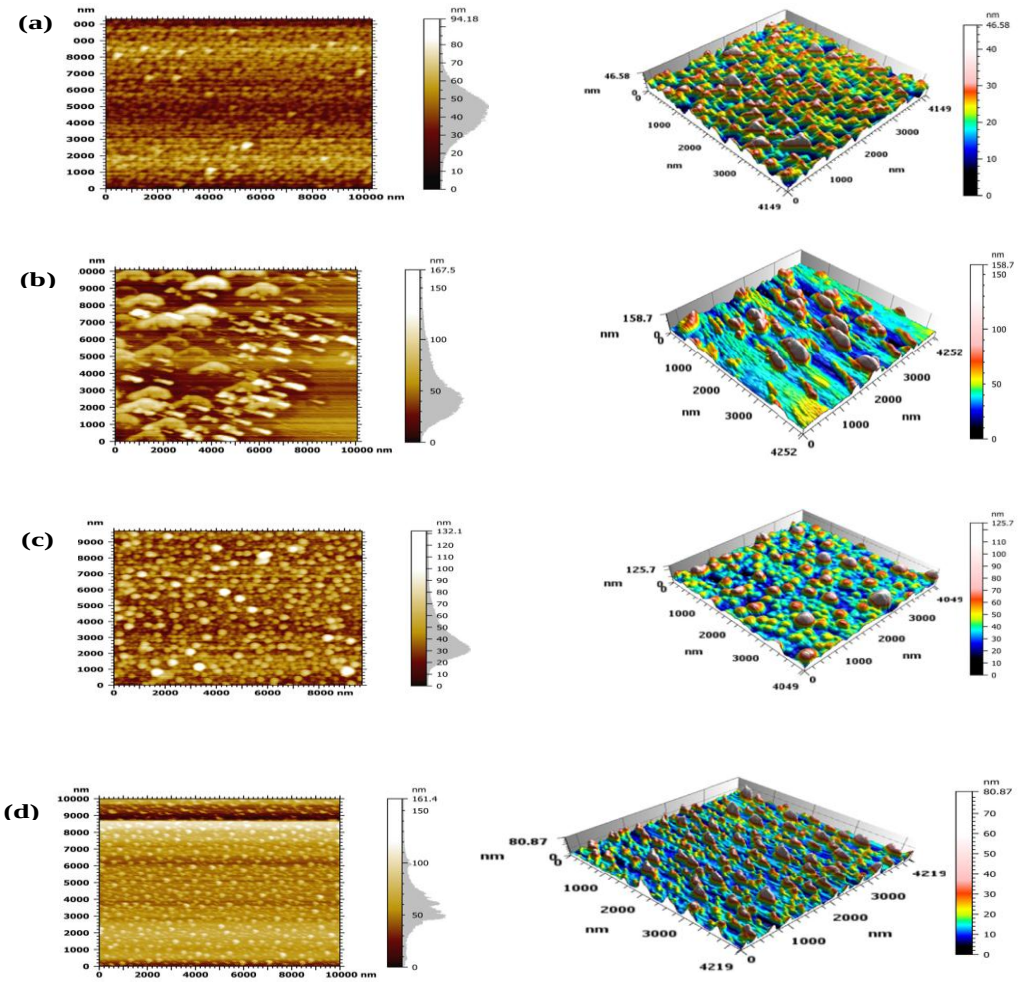

Fig. (4) AFM image of the prepared nanoparticles(a) Zn-Cr-LDH, (b) Zn-Cr-MMO-600, (c) Ni-AL-LDH, (d) Ni-AL-MMO-600.

4. Conclusion

In this paper, we reported that co-precipitation of Zn--Cr LDHs and Ni-AL LDHs nanoparticles and mixed metal oxidesMMO-600. The (XRD) results confirmed that Layered double hydroxides prepared have polycrystalline and the high crystallinity. The (FE-SEM) and (AFM) results assert the morphology of as-synthesized nanoparticles dependence on the annealing temperature, whereby the increase of the treatment temperature promotes porous surfaces and growth of the nanoparticles, eventually resulting in the development of well-defined faces.

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تخليق ومعالجة حرارية لجسيماتZn-Cr وNi-AL النانوية:التوصيف البنيوي والمورفولوجي

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مستخلص البحث:

تم تصنيع هيدروكسيدات مزدوجة الطبقات من Zn-Cr و Ni-AL بتقنية الترسيب المشترك. تم صنع منتج المعالجة الحرارية (MMO) عند (600) درجة مئوية. في نتائج (XRD) كانت جميع العينات متعددة البلورات. تُظهر نتائج (FE-SEM) أن المعالجات الحرارية اللاحقة لـ Zn-Cr LDHs و Ni-AL LDHs عند 600 درجة مئوية تؤدي إلى زيادة حجم الجسيمات النانوية إلى 51.53 نانومتر و 51.17 نانومتر على التوالي. تُظهر صور AFM توزيعًا جيدًا للجسيمات النانوية على السطح، ويزداد حجم الجسيمات بعد المعالجة الحرارية عند 600 درجة مئوية. توفر التكلفة المنخفضة بالإضافة إلى التحضير البسيط للهيدروكسيدات المزدوجة الطبقية المحضرة طريقة مثالية للحصول على جسيمات نانوية ذات أحجام بلورية صغيرة وبلورية عالية ومساحة سطح كبيرة.

الكلمات المفتاحية : الجسيمات النانوية؛ هيدروكسيدات مزدوجة الطبقات؛ أكسيد معدني مختلط؛ الترسيب المشترك؛ المعالجة الحرارية.