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SYNTHESIS OF THE COMPOUND $CdAl_2O_4$ BY THE METHOD OF SOL GEL AND STUDYING ITS STRUCTURAL PROPERTIES

Аннотация: В данной работе предложено синтезировать $CdAl_2O_4$ с использованием метода Золь-Геля, а температура синтеза установлена 600 °С. После подтверждения завершения процесса синтеза с помощью рентгеноструктурного метода. Индексы Миллера (hkl) рассчитывали для полученного соединения, и было обнаружено, что соединение, полученное методом золь-гель, кристаллизовалось в соответствии с гексагональной структурой кристаллизации. Пространственной группой симметрии является $R\bar{3}$. Диаграмма ИК-спектроскопии подтверждает, что искомое соединение было получено путем выявления пиков колебаний связи $Cd-O$ и $Al-O$ в кристаллической координации CdO_4 , AlO_4 .

Ключевые слова: солевой гель, смешанные оксиды, алюминат кадмия.

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1. Introduction: Methods of securing reliable and clean energy supplies are among the most important methods that scientists and researchers have taken care of in the 21st century because of their great importance in our daily, environmental and economic lives as well as on human health. Although the current energy sources meet our energy needs in the near future, they need a tremendous cost and emit health harmful gases, which are depleted sources. Therefore, the severe shortage in these energy sources has prompted scientists and engineers to exploit new technologies to search for sustainable, clean and highly efficient energy. Metal oxides are one of the most important categories used as a source of alternative energy, especially mixed oxides For example, metal oxides ($\text{LiO}_2\text{-ZnO}_2$) are used as rechargeable batteries and SnO_2 oxide is used in the manufacture of solar cells [1] , but unfortunately there are many reasons that hinder the spread of these technologies due to their high cost, insufficient duration and operating problem. Therefore, scientists seek to explore materials that reduce their cost and increase their efficiency. Therefore, mixed metal oxides have been synthesized, which often have properties that exceed pure oxides. Attention has been paid to synthesizing methods the Spinel oxides , Which attracted growing attention worldwide for its low cost and great use as an environment friendly technology Such as cadmium aluminate oxides CdAl_2O_4 formula which have multiple applications such as semiconductors and remote sensing devices [2][3].

2. Study the crystalline structure of cadmium aluminate oxide:

As it was mentioned in some reference cards that cadmium aluminate oxide can crystallize according to two types of crystallization

2.1. Cubic structure: The card with the number 5910208 in the COD database stated that the compound can crystallize according to the cubic crystal structure with a vacuum symmetry group $Fd-3m$ and has a crystal grid constant $a = 8.07800 \text{ \AA}$ [4].

2.2. Hexagonal crystal structure: There are other reference cards in the database COD number 96-722-7917. It was mentioned that the compound crystallizes according to the trigonal crystal system (hexagonal axes) with a space symmetry group $R3^-$ and has the following crystal grid constants ($Z = 18$ $a = 14.22100 \text{ \AA}$ $c = 9.57330 \text{ \AA}$) which have a density of 4.10600 g / cm^3 . The same structure was mentioned in another reference card found in ICDD database No. 00-022-0119 [5].

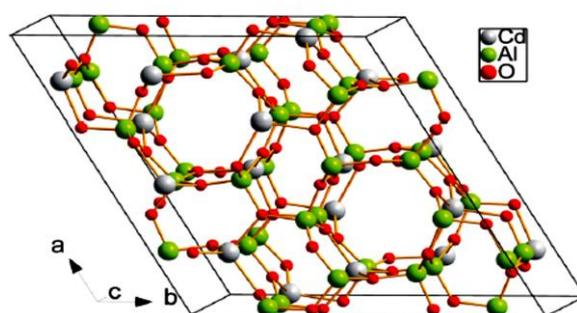


Figure 1. CdAl_2O_4 Crystalline Oxide Structure

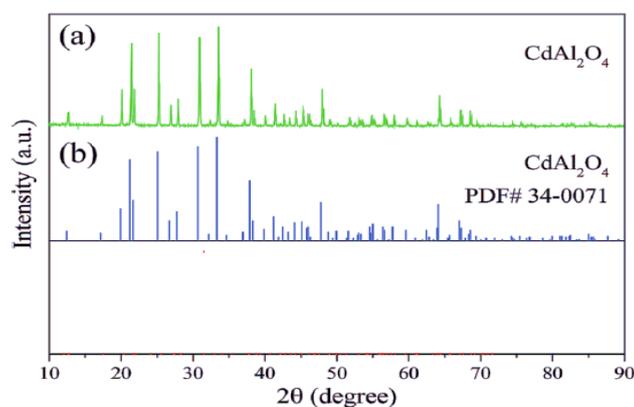


Figure 2. XRD patterns of CdAl_2O_4 Oxide

The following table shows the values of the diffraction angles according to the reference cards mentioned above.

3. Reference study: Mixed metal oxides play an important role in the academic field as well as industrial research and constitute the largest family of catalysts used in heterogeneous catalysis as the physical and chemical properties of mixed oxides such

as their catalytic activity and electrical resistance depend on the method of preparing the oxide, the calcination temperature and the raw materials used in the preparatory [2]. And many scientists are interested in studying cadmium aluminate oxide because it has good chemical and physical properties and most studies have shown the importance of this oxide as a photocatalyst for many important interactions. After conducting a reference study of this compound it was concluded that the synthesis of this compound in a solid-state method is difficult, due to the large spacing between the halves. The ionic diameters of Cd^{+2} / Al^{+3} (Cd (II) (0.92 Å) and Al (III) (0.50 Å)) where it was clarified that it is possible to synthesize $CuAl_2O_4$, $CoAl_2O_4$ and $MgAl_2O_4$, and for many ions whose half-ionic diameters are Close to aluminum ion as Al (III) (0.50 Å), (0.65 Å) Mg (II), Co (II) (0.65 Å) [4].

Many researchers have paid attention to the manufacture of cadmium aluminate oxides and we will mention some of these studies:

In 2010, researchers studied (M.N. Alaya a, *, A.M. Youssef b, A. Roumie c, R). Insert the effect of the preparation method and medium pH onto the synthesis of the compound $CdAl_2O_4$. As a starting point, the preparation of the hydroxides entering the composition of this oxide was studied. And by using different precipitation factors and different pH, precipitation was done by reacting the aluminum sulfate solution $Al_2(SO_4)_3 \cdot 16H_2O$ with both sodium hydroxide solution or ammonia solution. The same applies to cadmium sulfate and the following table shows the concentrations of the solutions used, the temperature and the precipitation factor for each sample prepared [2].

Table 1. Chemical and physical conditions used to prepare aluminum hydroxide $Al(OH)_3$

the sample	Concentration of solution $Al_2(SO_4)_3 \cdot 16H_2O$	Precipitation detector	Concentration of precipitation solution	PH	Temperature
AN6	1M	NaOH	3M	6	25 °C
AN7	1M	NaOH	3M	7	25 °C

AN7	1M	NH ₃ .H ₂ O	30%	7	25□
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Table 2. Chemical and physical conditions used to prepare cadimium hydroxide Cd(OH)₂

the sample	Concentration of solution CdSO ₄ .3H ₂ O	Precision detector	Concentration of precipitation solution solution	Temperature °C	PH
C8	1M	NaOH	2M	25□	8
C9	1M	NaOH	2M	25□	9

The aim of the researchers from these experiments was to demonstrate the effect of the change in the pH value and the different sedimentation factor on the thermal behavior of the sample. Then the ACM samples were prepared by the method of solid synthesis by mixing the oxides resulting from incineration of samples C8 with AN7 according to the following molar ratios (Al / Cd = 1: 0.25, 1: 0.5, 1: 1, 0.5: 1 , 0.25: 1)

- ❖ Also preparing the ACC samples using a common precipitation method by mixing a solution of Al₂ (SO₄)₃.16H₂O with a concentration of 1M with a solution of 1m CdSO₄.3H₂O with a concentration of 3M of sodium hydroxide and the pH = 8 and the molar ratios used were also (1: 0.25-1: 0.5 -1: 1-0.5: 1 - 0.25: 1) for Al / Cd respectively and after that the thermal behavior of samples was studied using DTA, TG device and the results are according to the following table

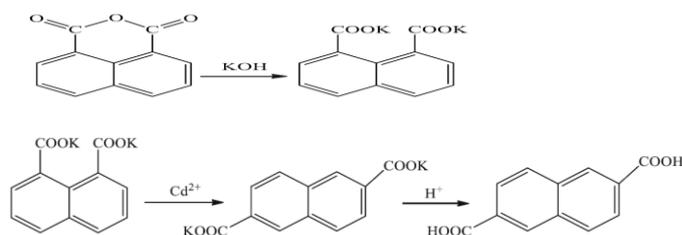
Table 3. Thermal behavior of the prepared samples

the sample	Temperature °C	Explanation	Type
ACM	100	4% Removing physical water molecules	Endo
	293	28% Dissolve chemical water molecules	Endo
ACC 1:0.25	900-920	9% Loss of the hydroxide group on the surface of the oxide	Endo
ACM	100	7% Removing physical water molecules	Endo
	296	10% Dissolve chemical water molecules	Endo

1:0.5	900	11% Hydroxides converted into oxides	Endo
	627	Cadmium aluminate is formed	Exo
ACM	245	5% Removing physical water molecules	Endo
0.5:1	276	21% Dissolve chemical water molecules	Endo

Thus, scientists proved that the thermal behavior of hydroxides and oxides relates to the sedimentation factor, pH, method of preparation, and degree of incineration [2].

In 2016 researchers worked (W Wang, L Cai, Y Hong, Y Jiang, Ch Wang) by their study on comparing the catalytic activity of pure oxide CdO with the catalytic activity of mixed oxide CdAl₂O₄. The stimulus experiment was the reaction of converting dipotassium 1,8-naphthalenedicarboxylate into 2,6-Naphthalenedicarboxylic acid which is the raw materials used in the manufacture of PVC and polyethylene, which have important characteristics such as tensile strength and elongation. The reaction scheme is shown by the following equations[3].



Scheme 1. Diagram of the reaction reaction of converting 1,8-naphthalenedicarboxylate into 2,6-Naphthalenedicarboxylic acid

Where the cadmium oxide was prepared by incineration the salt powder of Cd (NO₃)₂·4H₂O for 3h at 673k degree CdAl₂O₄ was manufactured by joint precipitation method by preparing 0.8 mol / l from a solution consisting of [X Cd (NO₃)₂·1-x Al (NO₃)₃] where (X = 0.85, 0.95, 1) and then 1mol of Na₂CO₃ was added to maintain the value of pH = 9. After that the precipitate was dried and burned at 673k for 3h. When the oxide manufacturing was finished, the resulting oxides were described by the following techniques XRD, SEM, FT-IR, XPS. By drawing an image of the samples using the SEM electron microscope it was found that the crystalline size of the sample CdAl₂O₄-0.95 is very small compared to the crystalline size of CdO oxide. Through all of the

above, it was shown that the catalytic activity of $\text{CdAl}_2\text{O}_4\text{-X}$ oxide when the value of $X = 0.95$ is much greater than the catalytic activity of the oxide CdO [3].

4. **The importance of research and its goals:** It was found that studying mixed metal oxides had more activity than pure metal oxides. In view of previous reference studies, it was concluded that the preparation of cadmium aluminate oxides is of great importance in applied fields such as solar cells and photocatalysts [8].

5. **The research aims to:** Preparation of the double oxide: $\text{CdO-Al}_2\text{O}_3$ using Sol-Gel method and studying the effect of temperature on the synthetic process

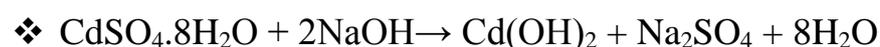
- Study the crystal structure of the resulting compounds
- Analyze the resulting samples with FT-IR and DTA spectroscopy

6. **Research materials:** High purity materials were used for analytical purposes:

Cobalt sulfate, NaOH 98%, Cadmium Sulfate $3\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$ (89%), Aluminum sulfate $\text{Al}_2(\text{SO}_4)_3 \cdot 16\text{H}_2\text{O}$ (97%), Substances used as a stabilizing agent (pectin - acetic acid - citric acid)

7. **The practical section:**

7.1. Sample preparation: In our research, we adopted the Sol-Gel method in order to obtain the compounds required for research, as this method is characterized by giving it a better mixture of homogeneity compared to other synthetic methods and requires low temperatures. The calculations necessary for preparation were performed based on the following equations:



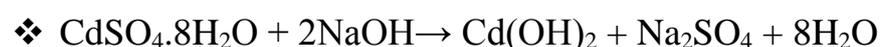
The final reaction is



Thus, we conclude that the stoichiometric ratio is appropriate (1: 1) with respect to

$\text{CdSO}_4 \cdot 8\text{H}_2\text{O} / \text{Al}_2(\text{SO}_4)_3 \cdot 16\text{H}_2\text{O}$. Therefore, the metal hydroxides entering the oxide composition were prepared according to the following:

7.1.1. the preparation of metal hydroxide $\text{Cd}(\text{OH})_2$: This hydroxide is prepared from the stoichiometric proportions determined by the reaction equation:



We prepared aqueous cadmium sulfate solution with a concentration of (0.1M) and a solution of sodium hydroxide with a concentration of (0.2M). The following table shows the masses of the materials according to the stoichiometric proportions determined by the reaction equation:

Table 4. weights of the raw materials necessary to prepare the compound Cd(OH)_2

Cd(OH)_2	
$\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$	2.6443g
NaOH	0.8000g

We took the weights specified in the table and solution each of them in (100mL) distilled water with stirring until complete dissolution without the need for heating. After that we added sodium hydroxide solution to cadmium sulfate solution in batches with continuous stirring by magnetic drive and after finishing the addition we got a deposit White and then filtered by centrifuge and separated. we wash the resulting precipitate several times with distilled water .

7.1.2. Preparation of Aluminum Hydroxide Al(OH)_3 : The same steps were followed by preparing the previous hydroxide where the reaction equation was



Where we prepared the aqueous aluminum sulfate solution with a concentration of (0.1M) and a solution of sodium hydroxide with a concentration of (0.6M), according to the ratio mentioned earlier, where it became clear that 2mol of aluminum hydroxide should be taken and the following table shows the masses of materials according to the stoichiometric proportions determined by the reaction equation:

Table 5. weights of the raw materials necessary to prepare the compound Al(OH)_3

Al(OH)_3	
$\text{Al}_2(\text{SO}_4)_3 \cdot 16\text{H}_2\text{O}$	6.4979g

NaOH	2.4g
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7.1.3. Mixing the prepared hydroxidine with the presence of the stabilizer:The stability and stability factor of Joomla were tested using three different stabilizers (vinegar acid - citric acid - pectin).

We divided the sediment evenly into 12 cylinders of equal size after each of the following cylinders was placed in the following solutions.

Table 6. Concentrations of stabilizer solutions used as stabilizer

Stability detector	Acetic acid				Pactin				Citric acid			
	0.05	0.1	0.2	0.3	0.05	0.1	0.2	0.3	0.005	0.025	0.05	0.1
Concentration of solution M	0.05	0.1	0.2	0.3	0.05	0.1	0.2	0.3	0.005	0.025	0.05	0.1
Volume of solution (ml)	30	30	30	30	30	30	30	30	30	30	30	30
No.	1	2	3	4	5	6	7	8	9	10	11	12

We left the previous cylinders for 72 hours, after shaking them evenly, and we found that the most appropriate concentration for the vinegar acid stabilizer is 0.05M, but with this the stability ratio was not satisfactory.

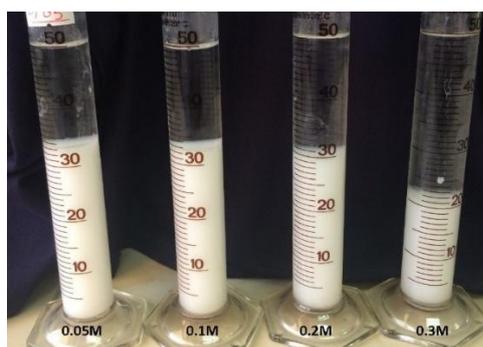


Figure 3. The CdO-Al₂O₃ Prepared Substance with the Acetic Acid Stabilizer

With regard to citric acid, all previous concentrations were inappropriate, where heterogeneity of the sentence was observed, one part of the precipitate was completely dissolved and the other part was deposited.

The percentage of separation increases with the concentration of the stabilizer, and this is shown in the following image:

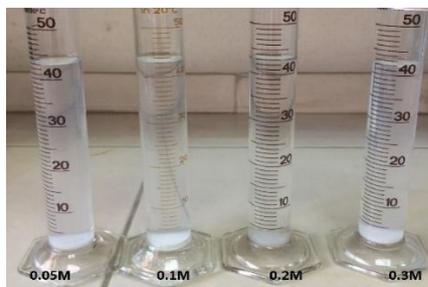


Figure 4. The CdO-Al₂O₃ prepared system with the presence of the citric acid stabilizer. As for the pectin solution, the most appropriate concentration for the stability of the system was 0.1M. However, it was noted that homogeneity and stability increase when adding drops of vinegar acid, which led us to use a mixture of vinegar acid and pectin as an ideal fixator for this system.



Figure 5. CdO-Al₂O₃ Prepared by Pectin Fixer

Then we prepared a new sample by following the same steps and leaving the hydroxide mixed with the stabilizer for a whole day, after which the formed precipitate was filtered and dried at 105 °C for 3h, after which the precipitate was thoroughly ground and the precipitate section was divided into several sections to perform the required analyzes on it.

The second stage of the work included placing the previous dried sample in high temperature tolerant ceramic crucibles of around 1200 °C with a view to burning them at different temperatures (1100 °C, 600 °C, 400 °C, 200 °C), with care being taken to monitor the synthetic process by obtaining a ray diffraction scheme. X-ray at different temperatures and compare the resulting charts with X-ray diffraction schemes for the primary compounds. Incineration has continued at each temperature for a period of time ranging from 4 hours, to know the best degree of synthesis.

8. Results and discussion:

8.1. Study the thermal behavior of vehicles using the differential thermal analysis device: This technique is based on the fact that when the compound is heated, it is subject to chemical and physical reactions and changes that involve absorption Or spread the heat. The following spectrum shows the thermal behavior of the sample before incineration:

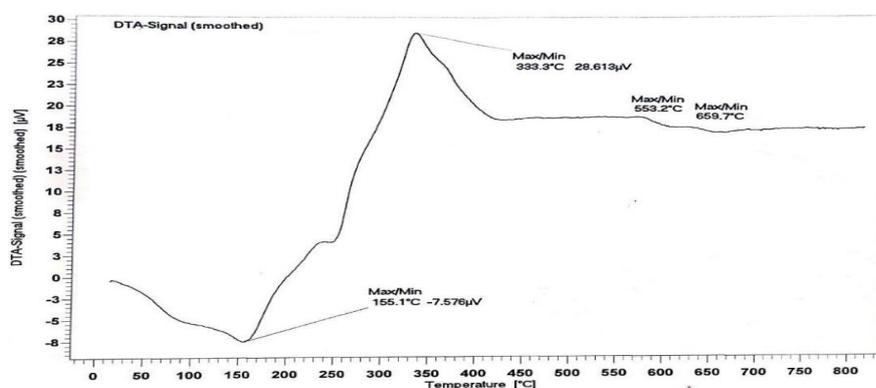


Figure 6. DTA spectrum for the sample prepared before incineration

The following table explains the DTA spectrum absorptions of a sample of cadmium aluminate

Table 7. DTA spectrum absorptions for a sample of CdO-Al₂O₃ cadmium aluminate

Explanation	type	Mass loss in percent	DTA Peak
Removing physical water molecules	Endo	%4.89	155.1 □
Beginning of oxide formation CdO-Al ₂ O ₃	EXo	%3.05	333.3 □
Starting crystallization of the compound	EXo	4.73	553.2 □
		5.53	659.7

We notice from the chart that the compound began to form at a degree of 333.3 °C and that it began to crystallize at a degree of 659.7 °C

We note the continuation of the curve height to the degree of 800 °C, which indicates the stability of the compound and the continued formation of this degree To prove the

results and to know more information, we incinerate the sample at different temperatures and perform a phase analysis of the samples by x-ray.

8.2. X-ray phase analysis: Incinerated cadmium aluminate was analyzed at different temperatures by an X-Ray Powder Diffractometer from Philips-PW-1840. The following shows the x-ray diffraction diagram showing the values of (2θ) and the peak intensity of the cadmium aluminate compound prepared by the Sol-Gel method. The distance between the crystalline levels (d) was calculated by the values of the diffraction angles. By a well-known relationship of Bragg ($n\lambda = 2d \sin \theta$). Figure (11) shows the incomplete X-ray diffraction diagram of the incomplete synthesis in the CdO- Al₂O₃ system at the degree 200 °C

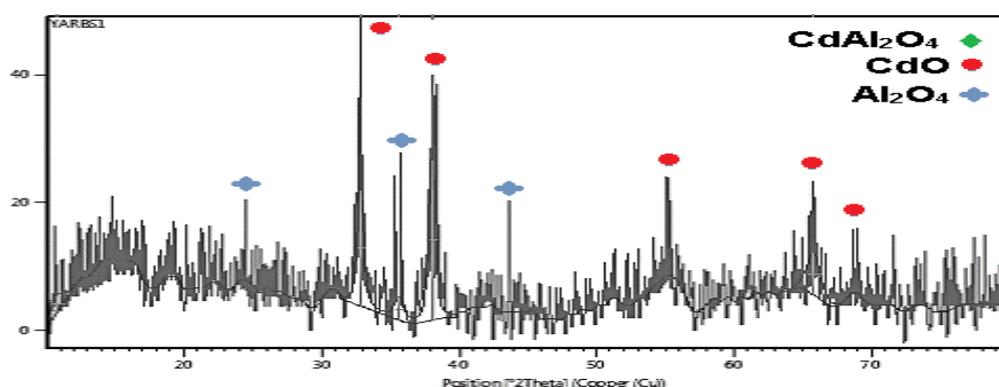


Figure 7. X-ray diffraction diagram for incomplete synthesis in the CdO- Al₂O₃ system at the degree 200 °C

We notice from the previous diagram the appearance of peaks to the primary oxides entering in the desired compound form, such as the peaks (43.55-35.9800-24.754) attributed to Al₂O₃ aluminum oxide, based on reference card number JCPDS NO.10-0173[9]. And peaks (33.086-38.115-55.278-65.937-68.647) belonging to the CdO cobalt oxide based on reference card number JCPDS NO. 5-0640.

We note that no peak appears to belong to the required compound, and thus we conclude that at this temperature the compound did not start to form and this was expected, based on the results of the DTA spectrum analysis, so the incineration temperature was raised. And Figure (14) shows the X-ray diffraction diagram of the incomplete synthesis in the CdO-Al₂O₃ system at the degree 400 °C

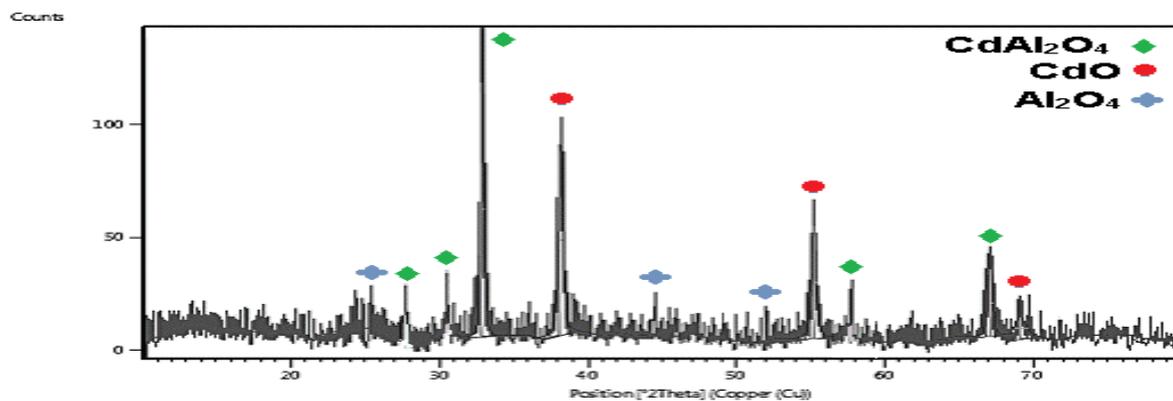


Figure 8. shows the X-ray diffraction diagram of the incomplete synthesis in the sentence CdO-Al₂O₃ at 400 °C

From the previous scheme, based on the reference card of the cadmium aluminate Compound No.COD (PDF 96-722-7917) Note the appearance of new peaks belonging to the compound required to be synthesized It is shown in the following table, with another peaks still attributed to the primary oxides

We notice from the table the appearance of six peaks attributed to the required compound with different intensities, with the value of the base peak in reference to the base peak in the prepared sample. And we notice the emergence of peaks to the raw materials mentioned in the previous paragraph, and this is evidence that the compound here began to form, but it was not completely synthesized and thus we conclude that this degree is the beginning of the formation of the required compound and therefore we raised the incineration temperature to 600 °C. And Figure 15.shows the X-ray diffraction diagram of the CdO-Al₂O₃ system at 600 °C

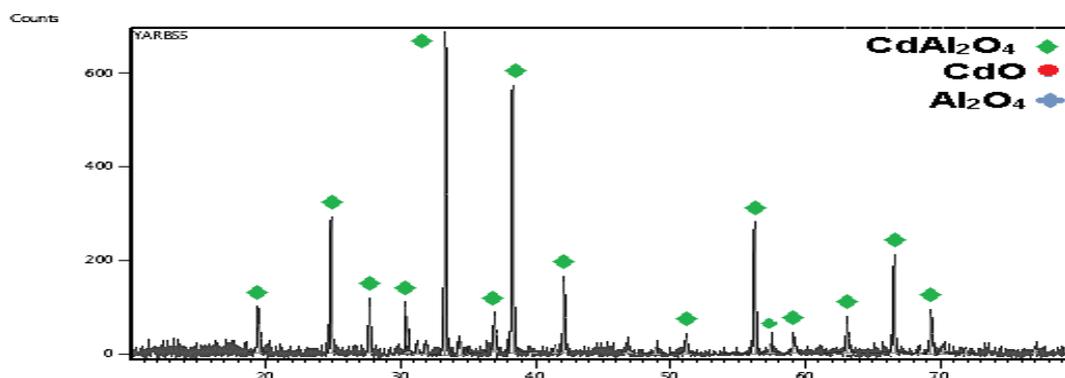


Figure 9. shows the X-ray diffraction pattern in CdO-Al₂O₃ at 600 °C

Table No. (11) shows the values of diffraction angles and distances between the crystalline levels and the Miller indices of the cadmium aluminate compound prepared at 600 °C

Table No. 8. The highest intensity peaks in the X-Ray diagram of CdO-Al₂O₃ at the 600 °C degree with the hexagonal crystal structure.

No	2 θ synthese	I/I ₀	θ	D	1/d ²	Hkl	a(A°)
1	19.7945	20	9.89725	4.481451	0.049792	102	14.21985
2	24.933	45	12.4665	3.568279	0.078538	220	14.27133
3	27.9	20	13.95	3.195187	0.097951	003	0
4	30.487	20	15.2435	2.929693	0.116508	113	14.50351
5	33.304	100	16.652	2.688042	0.138397	410	14.222
6	37	15	18.5	2.427572	0.16969	322	14.15764
7	38.445	80	19.2225	2.339588	0.182693	330	14.03577
8	42.219	35	21.1095	2.138767	0.218612	214	14.35468
9	51.37	10	25.685	1.777201	0.316612	440	14.21583
10	56.201	40	28.1005	1.635346	0.373922	710	14.25483
11	57.405	10	28.7025	1.603875	0.38874	6	0
12	59.1	10	29.55	1.561855	0.409939	116	14.15788
13	63.163	15	31.5815	1.470812	0.46226	614	14.08484
14	66.5	35	33.25	1.404869	0.506674	633	14.32616
15	69.214	20	34.607	1.35626	0.543644	642	14.22989

From the previous figure, we notice that this degree is the best in artificiality, as the synthesis of this compound was completed, and nine peaks to the required compound appeared sharply and with great intensity, and compared to the values in the resulting chart with the values of reference cards No. ICSD 61612 (PDF 01-078-0711) it was found that all values It is due to the cadmium aluminate oxide compound and the weak deviations in the values are due to the different method of synthesis. The compound was prepared in the reference by the method of solid synthesis by mixing the CdO-

Al₂O₃ and incineration at 900 °C and re-incineration at 1700 °C. As for our research, the synthesis was done using a sol-gel method using the salts of the minerals involved in the reaction and with the presence of a stabilizing agent as mentioned previously. At the end of the current calculations, it was found that the compound corresponds to the hexagonal symmetry pattern, and all the values of Miller indices for all summits are consistent with this pattern of crystallization that is consistent with the following relationship

$$\frac{1}{d^2} = \frac{4}{3} \frac{h^2 + k^2 + hk}{a^2} + \frac{l^2}{c^2}$$

If we look at the pattern of Miller indices (hkl) we notice that they fulfill the following condition: **h-k+l= 3n**. This condition corresponds to the symmetry group R3 of the hexagonal cell. The mean was calculated for each constant of the crystal grid constants a and c: a = 14.2279 (Å⁰) , c= 9.6044 (Å⁰). The base cell size was calculated and was **V= 1683.0(Å⁰)³**

When incineration to high temperatures 1100 °C, we notice that the compound appeared to dissolve, as many peaks appeared, and the peak base appeared at 2θ = 38.3646, which may return to the phase shift of the aluminum oxide. The following figure shows this.

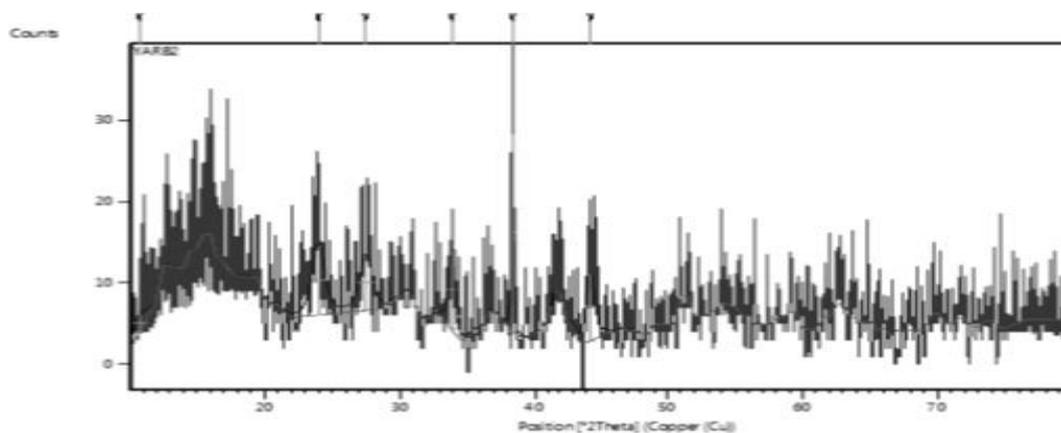


Figure 10. shows the X-ray diffraction pattern in CdO-Al₂O₃ at 1000 °C

8.3. Study of the resulting compounds using the infrared spectrum: Infrared spectra were withdrawn onto a Jasco-FT \ IR -5300 type device using KBr potassium bromide as an extension agent.

Figure 10. shows the infrared spectrum of the sample of cadmium aluminate burned at 600 °C.

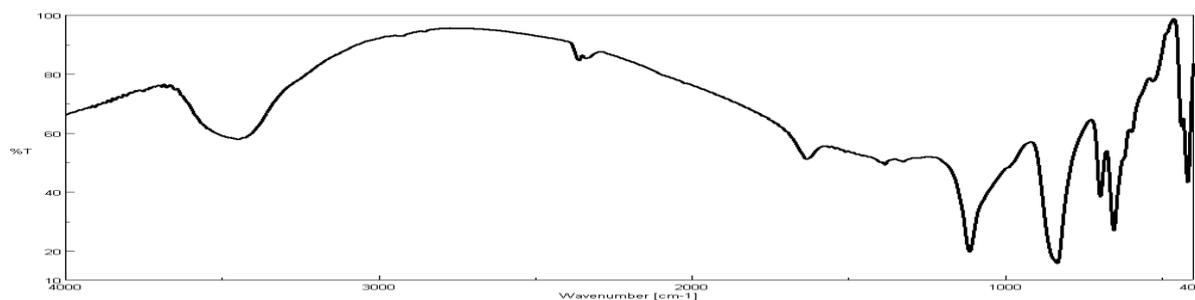


Figure 9. shows the infrared spectrum of the cadmium aluminate sample burned at 600 °C. As the spectrum shows that there are four absorption values, the following table explains the results.

Wave number Cm^{-1}	Vibration pattern
3448	Vibrating elongation of a bond O-H
1635	The bending vibration of water molecules within the crystalline structure
834	Vibrating elongation of a bond Cd-O
622-635-697-527-437	Vibrating elongation of a bond Al-O

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