Study the Structure, Morphology and Vibration Modes for K_2CrO_4 and $K_2Cr_2O_7$

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Abstract

In this work, the X-ray diffraction (XRD) measurement have been done to study the structure of K_2CrO_4, and $K_2Cr_2O_7$ powders.

The morphology of K_2CrO_4, and $K_2Cr_2O_7$ thin films has been studied by using Atomic Force Microscopy (AFM) technique.

Modes of vibrations for both K_2CrO_4, and $K_2Cr_2O_7$ compounds were measured and discussed using Fourier transform infrared spectroscopy (FTIR). [DOI: 10.22401/JNUS.20.2.09]

Keywords: structural, morphological, vibrational.

1. Introduction

1.1 Potassium chromate K_2CrO_4:

It is an inorganic solid compound, which has a yellow color for the potassium salt of chromateanion. It is known as a laboratory chemical material, whereas sodium chromate is an important in industrial material [1][2].

1.2 Potassium dichromate K2Cr2O7:

Potassium dichromate is one of the crystalline inorganic chemical reagent.

Hexavalent chromium compounds are harmful to health. $K_2Cr_2O_7$ is widely used in laboratories and industry as an oxidizing agent because it is not deliquescent. Potassium dichromate looks very bright and red-orange color [3][4].

Table (1) shows the physical and chemical properties of both K_2CrO_4 and $K_2Cr_2O_7$ compounds.

Table (1)
Shows physical and chemical properties for K_2CrO_4, $K_2Cr_2O_7$ [1-4].

physical and chemical properties	Potassium chromate	Potassium dichromate
Molecular Formula	K_2CrO_4	$K_2Cr_2O_7$
Molecular Weight	194.19 g/mol	294.18 g/mol
Physical State	Poly crystalline powder	Poly crystalline powder
Appearance	Yellow coloured powder	Orange coloured powder
PH (5%, 20 ^{0}C)	8.6 – 9.8	3.7 - 4
Density	2.73 g/cm^3	2.676 g/cm^3
Melting Point	968 ^{0}C	398 ^{0}C
Boiling Point	1000 ^{0}C	500 ^{0}C
Solubility in Water (20^{0}C)	629 g/L	125 g/L
Solubility in Alcohol	Insoluble	Insoluble
Refractive Index (n_D)	1.74	1.738

2. Expermantal procedure

2.1 Prepartion of K_2CrO_4, $K_2Cr_2O_7$ thin films:

2.1.1 Substrate preparation:

For spin coating (K_2CrO_4) or ($K_2Cr_2O_7$), thin films have used glass substrates. At the beginning, the substrates have been cleaned by diluted hydrochloric acid (HCl), ethanol and distilled water, then using electrical oven supplied by (Jard) at temperature (80 C^0) to dry the substrate.

2.1.2 Film spin coating:

Potassium chromate powder or potassium dichromate powder is grinded with grinder to get fine powder then the ethanol is add to fin

powder and enter the precipitate solution into the ultrasonic vibration for (15) minutes to become homogeneous solution. Spin coating is applied by adding a drops of homogeneous solution by Pipette to glass slide to get thin film.

The summary of spin coating conditions is showed in Table (2).

Table (2)
Summary of spin coating conditions.

parameters	values
Speed	3000 (rpm)
Time	15 sec
acclr	5 sec
Temperature	25℃

3. Results and discussions

3.1 X- ray diffraction (XRD):

K_2CrO_4 powder is grinded with grinder to get fine powder in order to get perfect crystallinity of material as clarified in Fig.(1A). The characteristics peaks of X-ray diffraction pattern for K_2CrO_4 come at $2\theta=29.0750^o$, 29.9389^o, 20.8283^o which correspond to miller indices (211),(031),(111) respectively; according to the JCPDS no.15-365 data. And all the hexagonal phase (Orthorhombic structure) of K_2CrO_4 is appeared in all diffraction peaks. The high purity of K_2CrO_4 compound indicated disappearance peak of impurity.

The lattice spacing (d) could be calculated by using Bragg's law; to calculate (d) lattice spacing where $n_i=1$.

$$2d \sin\theta = n_i \lambda \quad \text{..... (1)}$$

where λ of the X-Ray wavelength is ($1.54\ A^0$), θ is the diffraction angle in degree and $n_i=1$.

Also the grain size (D) can be calculated by using Scherrer equation:

$$D=\frac{k\lambda}{\beta\cos\theta} \quad \text{..... (2)}$$

where k= is a constant ($0.89<k<1$), β=FWHM. (full width at half maximum) of the diffraction peak.

The particle size (D) is calculated using equation (2).The average crystallite size D has been calculated from equation (2) to be 12.95 nm.

$K_2Cr_2O_7$ powder is grinded with grinder to get fine powder in order to get good crystallinity of material as shown in Fig. (1B). The X-ray diffraction pattern shows characteristic peaks of $K_2Cr_2O_7$ at $2\theta=27.1046^o$, 25.7088^o, 24.4468^o which correspond to miller indices ($2\bar{1}1$),(210),($0\bar{1}2$) respectively according to the JCPDS no.0.27-0380 data. All the diffraction peaks refer to hexagonal phase (Anorthic Triclinic structure) of $K_2Cr_2O_7$ peaks of impurity which do not be observed, that refers to the high purity of $K_2Cr_2O_7$ compound.

The particle size calculated using equation (2) shows an average crystallite size D of 19.91 nm.

Table (3) shows XRD details for K_2CrO_4, $K_2Cr_2O_7$ powders.

Table (3)
XRD for K_2CrO_4 and $K_2Cr_2O_7$ powder.

Material	2θ (deg.)	Plane (hkl)	FWHM (deg.)	Grain size (nm)	d (Aº)
K_2CrO_4	29.0750º	211	0.26980	15.21	3.06876
	29.9389º	031	0.36830	11.168	2.98215
	20.8283º	111	0.32350	12.489	4.26140
$K_2Cr_2O_7$	27.1046º	$2\bar{1}1$	0.24850	16.448	3.28720
	25.7088º	210	0.25470	16.002	3.46242
	24.4468º	$0\bar{1}2$	0.14900	27.287	3.63823

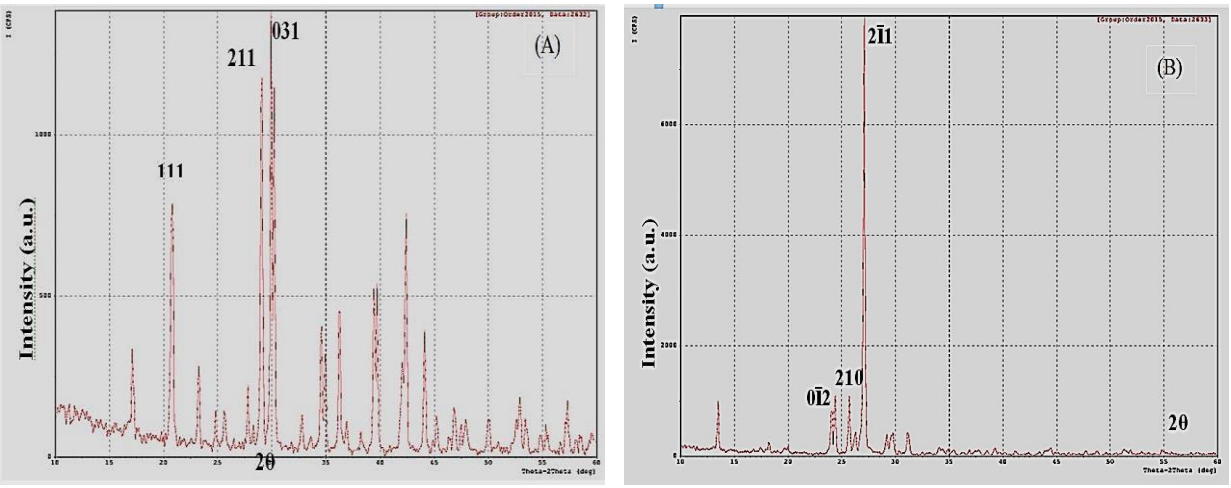
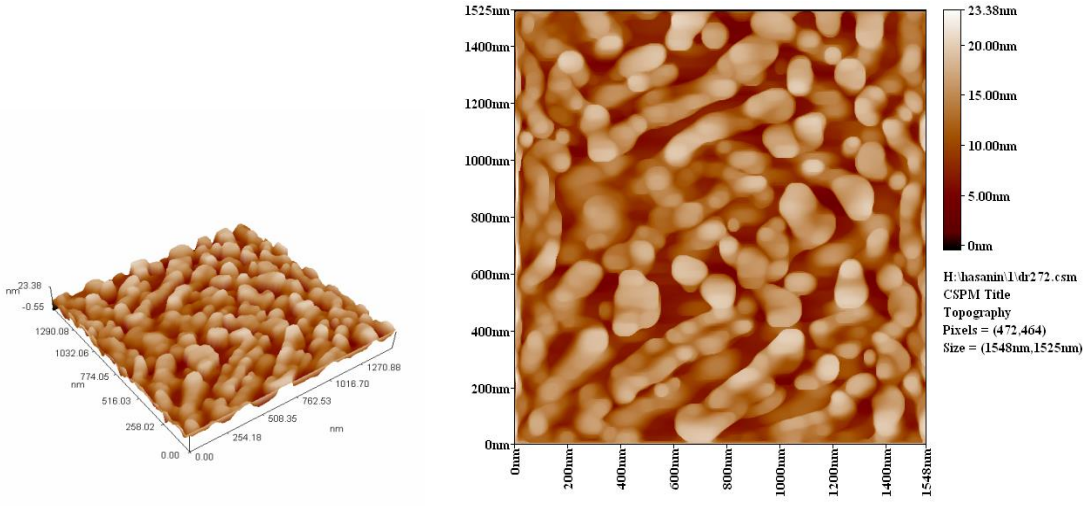


Fig.(1): The XRD spectra of the (A) K_2CrO_4, (B) $K_2Cr_2O_7$ powders.

3.2 Atomic Force Microscopy (AFM):

In sensing devices application the morphology plays an important role to the physical and optical properties. Fig.(2 A) refers to the 3D, 2D AFM images of the K_2CrO_4 thin film, while Fig.(2 B) refers to the 3D, 2D AFM images of the $K_2Cr_2O_7$ thin film.

(A)



(B)

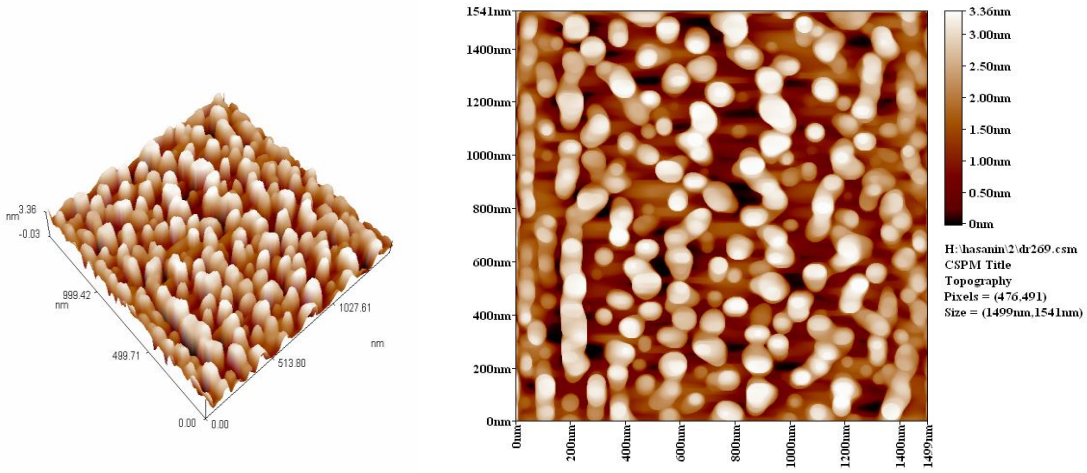
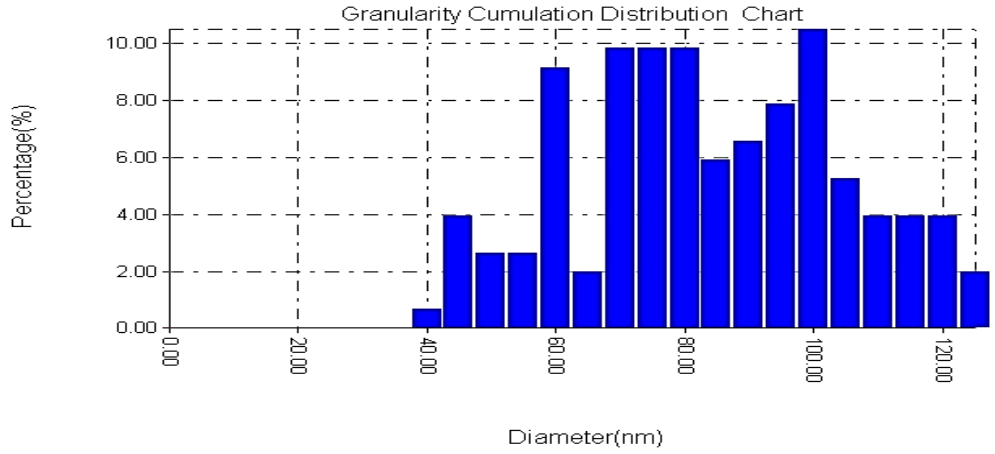


Fig.(2): Atomic force microscopy images at 3D and 2D of the (A) K_2CrO_4 thin film, (B) $K_2Cr_2O_7$ thin film.

Fig.(3) refers to the granularity cumulation distribution chart of the K_2CrO_4, $K_2Cr_2O_7$ thin films.

The calculated values of average grain size and roughness are shown in Table (4).

(A)



(B)

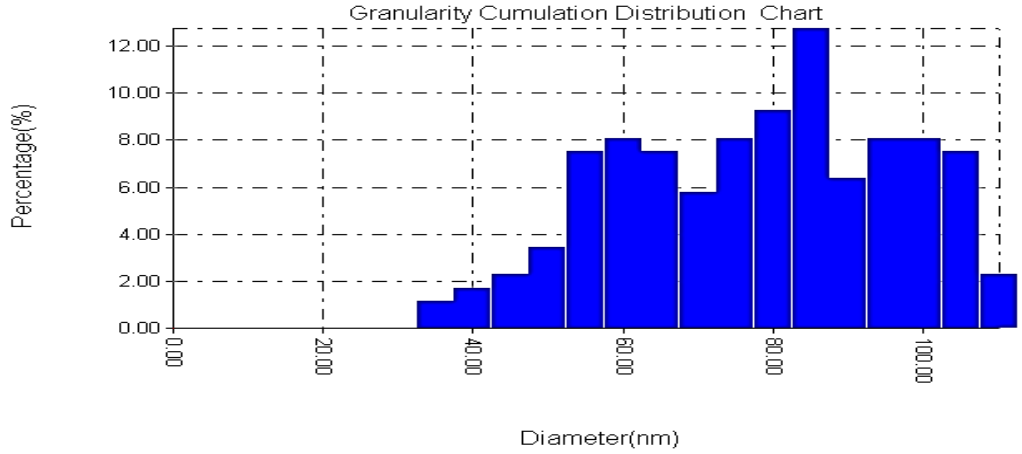


Fig.(3): Granularity cumulation. distribution chart of the (A) K_2CrO_4 and (B)$K_2Cr_2O_7$ thin films.

Table (4)
The value of average grain size and roughness.

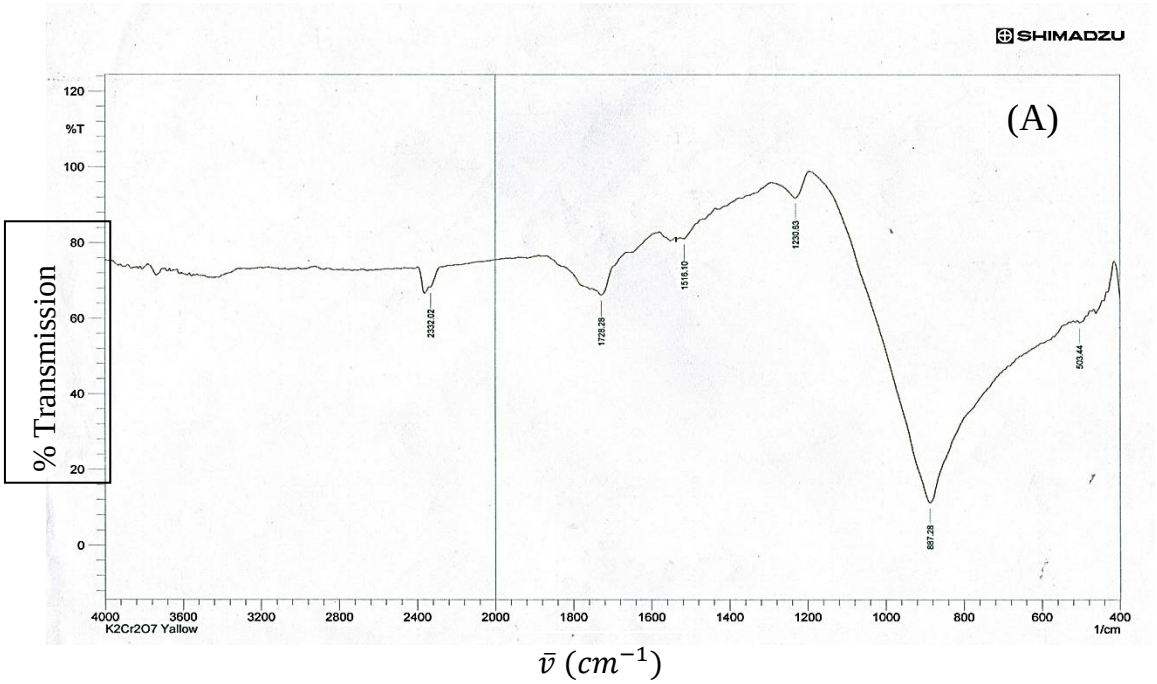
Material	Average. grain size (nm)	Roughness(nm)	Root,, Mean Square (nm)
K_2CrO_4	81.23	3.34	3.96
$K_2Cr_2O_7$	75.53	0.815	0.974

3.3 Fourier Transform Infrared Spectroscopy (FTIR):

Fig.(4A) shows the FTIR spectrum of K_2CrO_4 powder. The CrO_4^{-2} ion has tetrahedral structure belonging to T_d point group. This ion has four fundamental bands; υ_1 (symmetric stretching band), υ_2 (anti- symmetric stretching band), υ_3 (symmetric bending band), υ_4 (anti-symmetric bending band). The υ_1 and υ_2 vibrations are Raman active only (this is because there are only changes in magnitude or direction of polariziblity), while υ_3 and υ_4 vibrations are infrared active only (this is because there are changes in magnitude of dipole moment).

From the previous studies that a free chromate ion as a result of its T_d symmetry has four normal modes of vibration represented by A_1,E_1,T_1 and T_2 with frequencies υ_1(847 cm^{-1}), υ_2 (348 cm^{-1}), υ_3 (884 cm^{-1}), and υ_4 (368 cm^{-1}) respectively [5][6].

Fig.(4B) shows the FTIR spectrum of $K_2Cr_2O_7$ powder. The weak peak of $K_2Cr_2O_7$ at 555.52 cm^{-1} is due to Cr-O-Cr symmetric stretching vibration, while the very strong peak at 754.19 cm^{-1} is due to Cr-O-Cr anti symmetric stretching vibration, the medium strong peak at 891.14 cm^{-1} is due to $Cr\text{-}O_3$ symmetric stretching vibration (ms) and the very strong peak at 935.51 cm^{-1} is due to $Cr\text{-}O_3$ anti symmetric stretching vibration (vs) [7] [8].



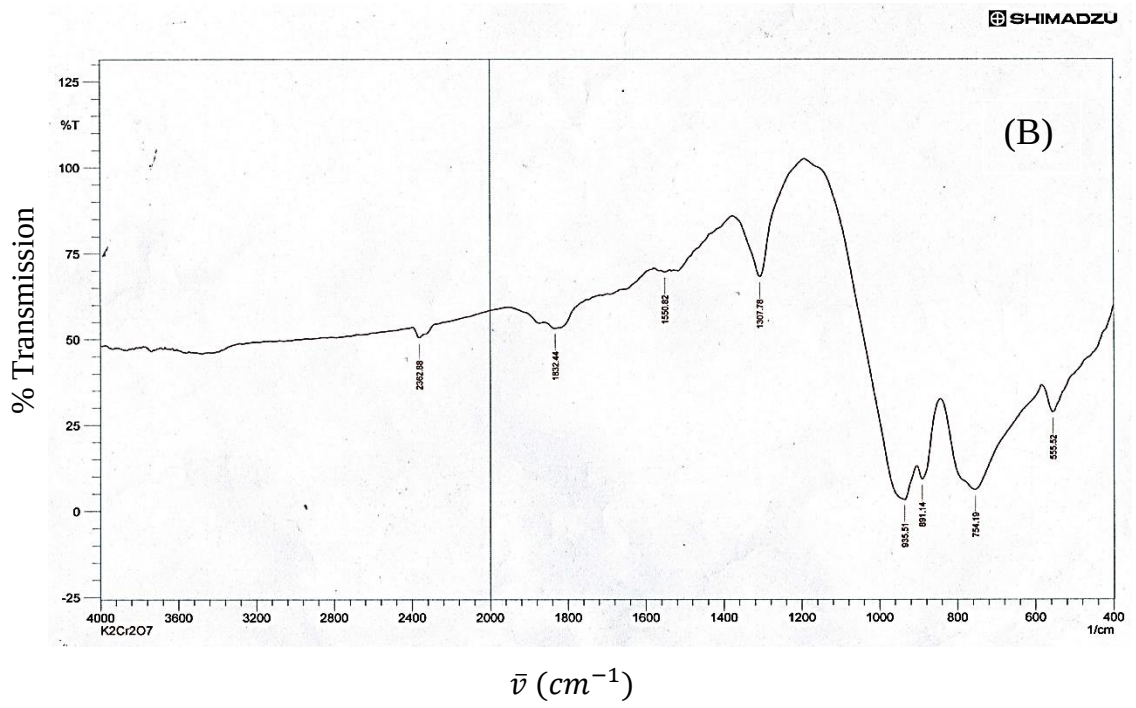


Fig.(4): Shows FTIR spectra for (A) K_2CrO_4 powder (B) $K_2Cr_2O_7$ powder.

4. Conclusions

1. The X-ray diffraction (XRD) results showed that K_2CrO_4 and $K_2Cr_2O_7$ nanoparticles powder have good crystallinity and in general the grain size of K_2CrO_4 compound was smaller than that of $K_2Cr_2O_7$ compound.
2. The Atomic force microscopy (AFM) results showed that K_2CrO_4 compound was more average grain size and roughness than that for $K_2Cr_2O_7$ compound.
3. FTIR spectra show absorption bands assigned both stretching and bending vibrations related to the two compound K_2CrO_4 and $K_2Cr_2O_7$.

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