Decomposition of Formaldehyde Using Vanadium Oxide Nanostructures as Catalysts

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A R T I C L E   I N F O

A B S T R A C T

The speed of formaldehyde decomposition has been studied using catalysts (vanadium nanoparticles, nano vanadium oxide with nano hydroxyapatite and vanadium oxide). The catalysts have been made with three different concentrations (100, 500 and 1000) ppm and at three different temperatures (25, 50 and 75) ° C. X-ray diffraction (XRD) and transmission electron microscope (TEM) images were used to study the structural and engineering properties of the prepared catalysts, which were nanostructured materials. The increase in the concentration of the catalysts and the temperature led to increasing the chemical reactions rate. However, it decreased the activation energy, which was calculated using the Arrhenius equation. The catalyst NV_2O_5 showed a better reaction rate compared to other prepared catalysts $100.1 \, x \, 10^{-3}$ and $112.5 \, x \, 10^{-3}$) for the catalysts such as nano vanadium oxide and nano vanadium oxide with nano hydroxyapatite, respectively.

1. INTRODUCTION

Over the recent years, the catalysts achieved a noteworthy development through many new applications. It is considered the main basis in the success and development of many new processes employed in several industrial fields, as it works to reduce waste and emissions in cars and fuel stations. Moreover, it is used in many of the most important interactions such as alkylation, pasteurization and oxidation reactions[1,2]. Nanomaterials have been used in several applications, including the medicine. Hydroxyapatite (HAP) is considered a form of apatite, which is a chemical material that similar to the acidic material in organisms in terms of chemical composition and phase. The pure hydroxyapatite is usually brittle; hence, its applications in research are limited. Several efforts have been made to improve its properties by controlling the particle size by making it within the nanoscale. Hydroxyapatite nanostructure is a biological substance used with the solid tissues of the body[3], water treatment and other applications[4].

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Formaldehyde is a colourless, extremely flammable gas at room temperature with a molecular formula CH_2O, which can be commercially obtained as a water solution of (20-50%) that is known as formalin. The formaldehyde oxidizes in the air under the light in carbon dioxide and it also reacts very quickly to hydroxyl to produce formic acid according to environmental conditions[5].

Nano-vanadium oxide has attracted considerable attention due to its chemical and physical properties and its great potential in the application of chemical catalyses such as electromechanical devices, sensors, electrochemistry, and photocatalytic activities. Vanadium oxide (V_2O_5) is one of the most stable compounds of vanadium oxides because its crystal structure consists of two-dimensional layers. Nano vanadium oxide can be used to convert energy such as chemical energy (fuel cells) and solar energy (photovoltaics) as well as thermal energy (thermal energy generation)[6,7]. It also showed excellent results when mixed with the molybdenum to improve the performance of negative lithium-ion batteries[8]. Many studies have been conducted to prepare catalysts via several

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methods and through using theirs in different applications. Danilevich et al.[9] studied the effect of the supporting of vanadia by (SiO_2, Al_2O_3, ZrO_2, and TiO_2) by fertilization method on the oxidation of formaldehyde to formic acid and characterized the catalyst using X-ray diffraction (XRD), X-ray spectroscopy (XPS), Raman spectroscopy, UV-Vis spectroscopy and temperature-programmed reduction (TPR). They reported that the activity of formaldehyde oxidation to formic acid depends on the vanadia structure. They have concluded that TiO_2 is the most suitable support for the formation of monolayer vanadium, which is the most effective in oxidizing formaldehyde to formic acid at 120ºC. Kaichev et al.[10] used the active compounds of the supported vanadium as an aid to methanol oxidation, the structural and the catalytic properties of the supported catalyst (vanadium oxide) with different oxides such as SiO_2, γ -Al_2O_3, ZrO_2, and TiO_2 were examined at temperatures ranging between (200-100)ºC. They concluded that the nature of the support determines the shape of the vanadium structure. The addition of SiO_2 and γ-$Al2O_3$ led to the formation of the best crystals of vanadium oxide. It also demonstrated that the vanadium oxide crystals are less active in the selective oxidation of methanol, and the polymeric forms are more active than monomer. Dwivedi et al. [11] examined the vanadium oxide loaded on alumina properties as a catalyst to oxidation of 2-Chlorotoluen(2-CLT) to 2-hlorobenzonitrile (2-CLBN). They concluded that the maximum productivity of (2-CLBN) reaches 76% at a temperature of 425ºC, and that the X-ray spectroscopy of the pure and consuming catalyst shows that the catalyst has decreased during the oxidation process.

2.   EXPERIMENTAL METHOD
2.1. Preparation of a Hydroxyapatite Nanoparticle (NHAP)

According to Orlovskii et al. [12], nano hydroxyapatite was prepared by adding calcium nitrate tetrahydrate ($Ca(NO_3)_2.4H_2O$) solution 0.65 M in drops to ammonium dihydrogen phosphate ($NH_4H_2PO_4$) 0.5M with continuous stirring at 80 ºC. The pH value was kept within 10 during the preparation. When adding ammonia at a concentration of 25%, the resulting precipitate is collected, filtered, washed with hot distilled water, and dried at 80ºC for 24 hours. Then, the precipitate is calcined at 400ºC for four hours under normal atmospheric pressure.

2.2. Preparation of Nano Vanadium Oxide

Vanadium oxide nanostructure was prepared according to Nalini and Babu [13,14], by heating ammonium Meta Vanadate solution 0.01M while stirring. Then the nitric acid (HNO_3) was slowly added with stirring (stirred for an hour and keep the value of the PH within the limits of (2 - 3). A precipitate is formed in red color, which is filtered and dried at 100ºC for 24 hours.

2.3. Preparation of Mix (NV2O5 and NHAP)

Two grams of NHAP were dissolved in 50 ml of NV_2O_5 solution with continuous stirring at 60 oC for half an hour, a light yellow precipitate is formed that collects the precipitate by filtering, dried at 80 ºC for 24 hours and calcification for 4 hours at 400 ºC.

3.   RESULTS AND DISCUSSION
3.1. Structural Properties

The structural properties of the prepared materials were studied by analysing X-ray diffraction performed with the SHIMADZU-6000 (X-ray powder Diffraction). The crystalline size was calculated using scherrer's equation[15];

$$D = \frac{k\lambda}{\beta \, Cos\theta} \dots \dots \dots (1)$$

where λ is the wavelength of x-ray, β is half at width full maximum, and k shape constant which is about 0.9.

Fig.1. represents the X-ray diffraction patterns of the prepared NV_2O_5 powder, the peaks of NV_2O_5 are located at 2θ = 25.04º and 27.73º in the orientation (001) and (120), respectively, as well as in the phase of (γ V_2O_5) at 2θ = 30.759º at (210) of an orientation with a crystalline size of approximately (27.5 and 4.8) nm as shown in table 1. This corresponds to the cards numbered (98 - 008 - 0594), (00 - 053 - 0538) respectively, the NV_2O_5 in both phases possesses an orthorhombic crystal structure is found. V_2O_5 accounted for approximately 22% of the amount of powder produced from the reaction, in addition to the presence of the $H_3O_9V_3$ phase which has the highest peak and the $H_xV_2O_5$ phase.
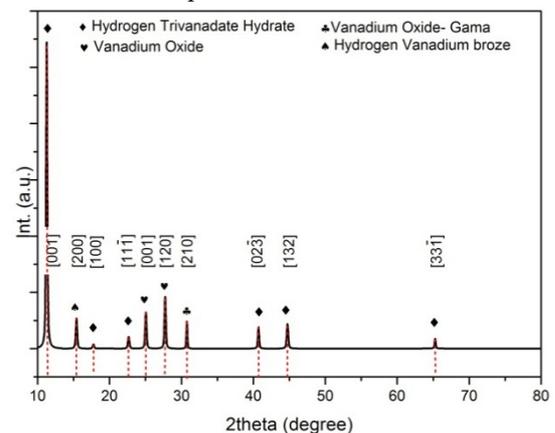
Fig.1 **:** X-ray diffraction pattern of the prepared NV_2O_5 powder.

The addition of vanadium oxide nanoparticle to nano-hydroxyapatite only led to 0.3% of the correlation in the reaction product represented by the appearance of the phase (Ca_5 (VO_4) OH) at the angle (2θ = 22.185º) and direction (111). The apatite ratio was estimated at about 72% of the reaction result, and additional phases of ($Ca_5(PO_4)OH$) and ($Ca_3(PO_4).XH_2O$) are observed As it is evident in Fig. 2 and table. 2.
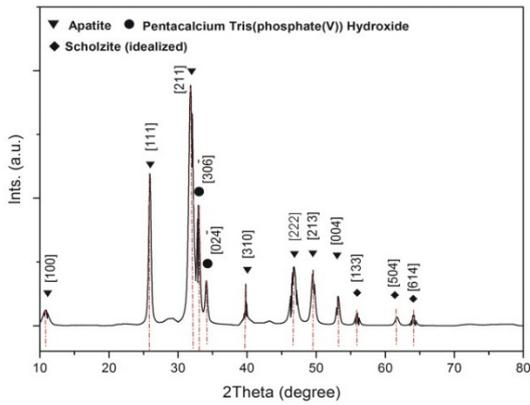
Fig.2: X-ray diffraction pattern of the mixture of vanadium oxide and prepared nano-hydroxyapatite.

3.2. Morphological Properties

The TEM images of the NV_2O_5 compound showed that there are cluster groups of particles with average size of 27 nm, as shown in Fig. 3.B, as well as the presence of plates of irregular shapes whose lengths range about 1.5 μm and a width of about of 670 nm as shown in Fig. 3.C. The apparent difference in the shapes of the prepared particles is a result of the phases formed in the reaction output, and this is confirmed by the difference in the observed peaks of the resulting phases in the X-ray diffraction model shown in Fig.1.
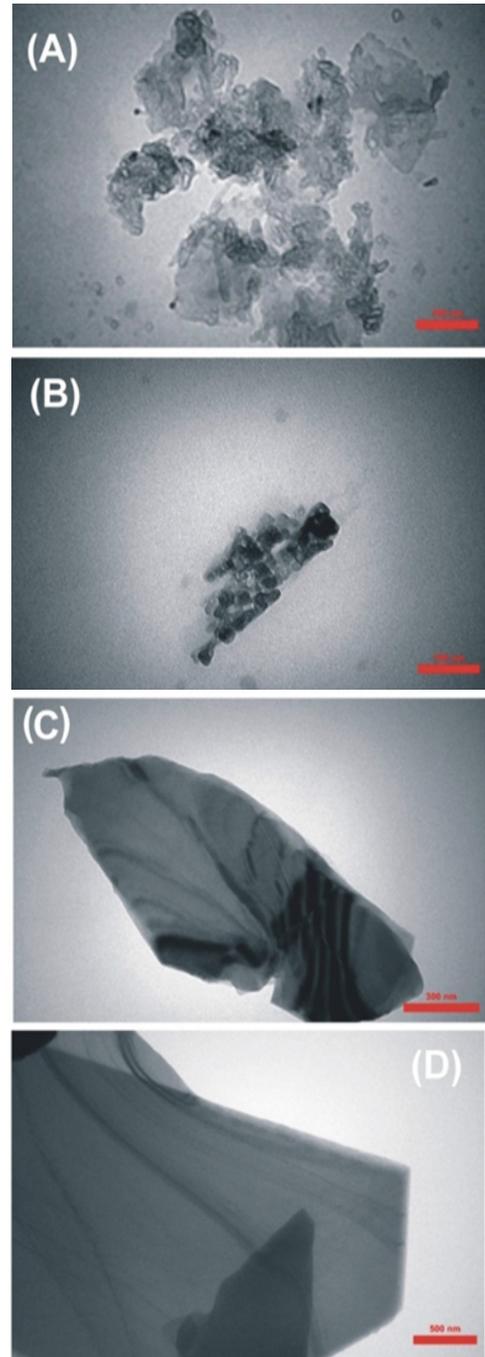
Fig.3: TEM images of vanadium oxide nanoparticle and the associated phases during the preparation.

Fig.4 shows the TEM images of the mixture of (NV2O5 and NHAP), as it shows the control of the petite particles that appear in the form of rods whose length ranges between 40 nm and 100 nm with diameters ranging between 25 nm and 75 nm, in addition to bars with a length of 0.7 μm and a diameter of about 75 nm, and cubic particles of average size of 10 nm.
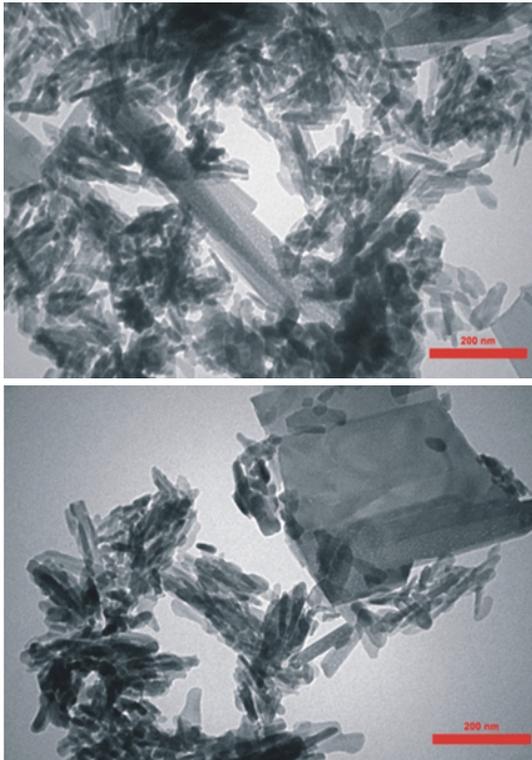
Fig.4: TEM images of NV2O5 and NHAP nanoparticles
and their associated phases during the preparation.

3.3. Kinetics of Reaction

The interaction speed varies according to the type of interaction and the number of molecules involved in it when the temperature is fixed. For the purpose of distinguishing between these reactions, the term reaction order has been used, which can be defined as the sum of the exponents to which the concentrations of the reactants are raised in the speed equation. Therefore, the law of reaction velocity depends on the reaction order [16]. In first-order reactions, the reaction velocity can be calculated using Eq. 2

$$r = k\,[C] \dots\dots\dots(2)$$

where r is the reaction velocity, k is the reaction velocity constant, and $[C]$ is the concentration. The reaction of all the prepared catalysts with formaldehyde is of the first order under all reaction conditions in terms of temperatures and the used concentrations as shown in Fig.s (5, 6 and 7).
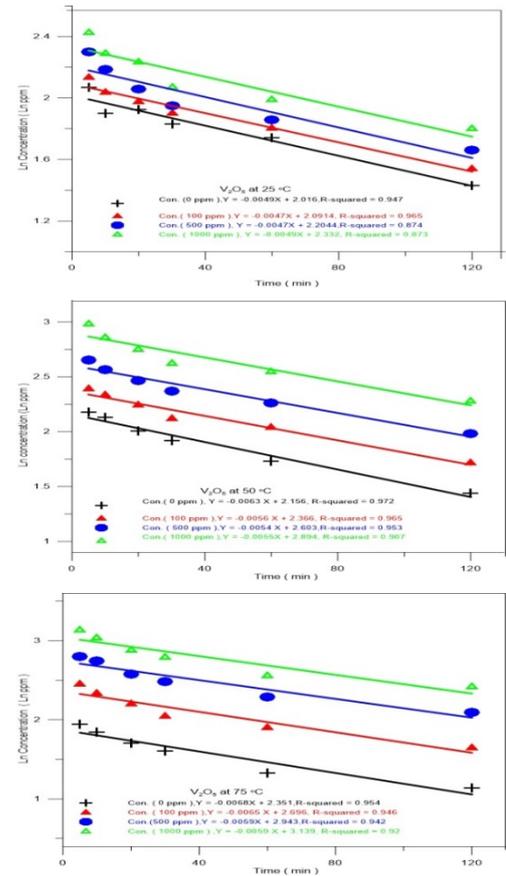
Fig.5: The relationship between (ln con.) and time using the catalyst (NV_2O_5) under the influence of temperatures (25, 50, 75) °C.
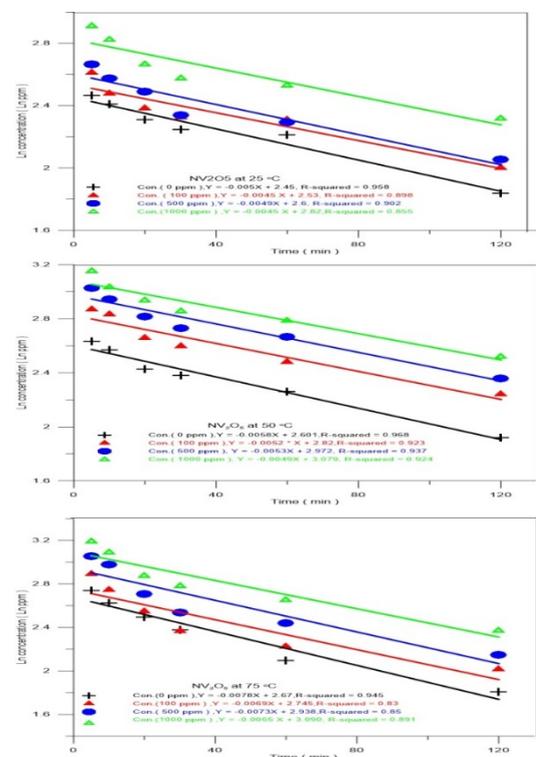
Fig.6 :The relationship between (ln con.) and time using the catalyst (V_2O_5) under the influence of temperatures (25, 50, 75) ºC

3.3.1. The Catalytic Agent (vanadium nanoparticle) (NV_2O_5)

The catalyst (NV_2O_5) was added in concentrations of (100, 500, 1000) ppm to formaldehyde solution at temperatures (25, 50, 75)°C. The reaction rate increases with increasing the temperature and concentration, where the highest reaction speed rate was (113.67×10^{-3}) mg /L.min at the concentration of 1000 ppm and a temperature of 75 °C.
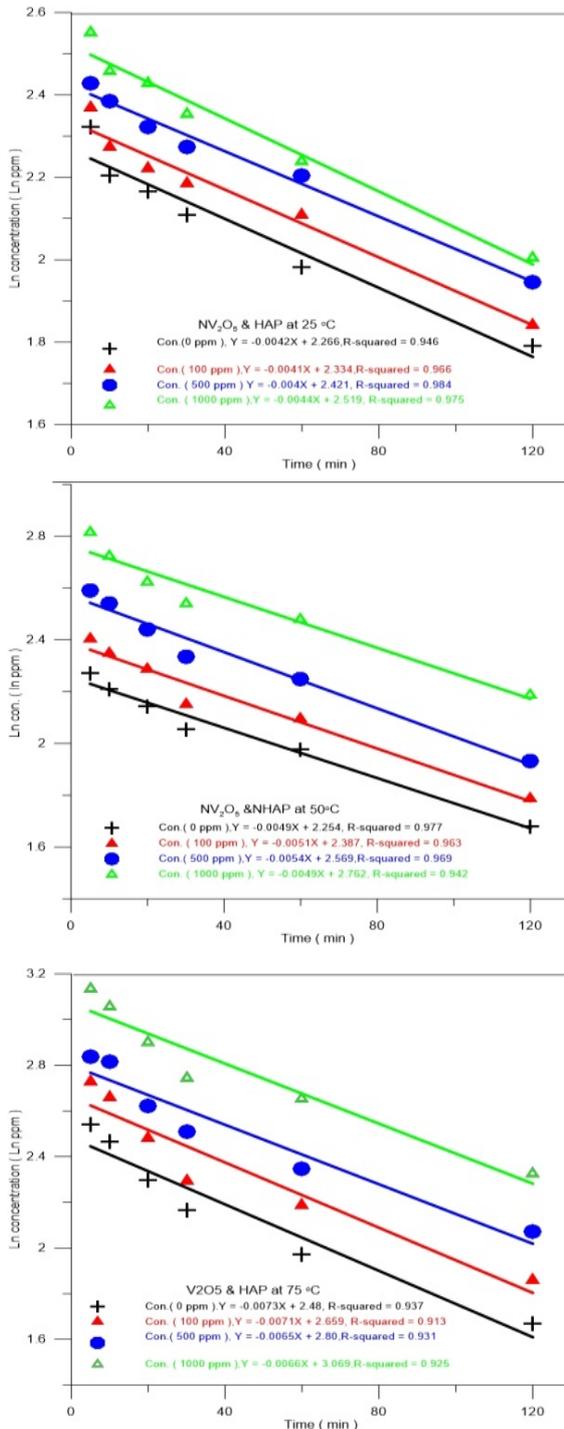
Fig.7: The relationship between (ln con.) and time using the catalyst (NV2O5 and NHAP) under the influence of temperatures (25, 50, 75) °C.

3.3.2.The Vanadium Oxide Catalyst (V_2O_5)

The catalyst (V_2O_5) was added in concentrations of (100, 500, 1000) ppm to formaldehyde solution at temperatures (25, 50, 75)°C. The reaction rate increases with increasing the temperature and concentration, where the highest rate of reaction speed was (100.1×10^{-3}) mg / L.min at the concentration of 1000 ppm and a temperature of 75 °C. Table.3 shows the values of the reaction rate of both NV_2O_5 and V_2O_5 with formaldehyde according to the concentration and temperatures.

3.3.3.The Catalyst of The Vanadium Oxide Mixture Nanoparticle with Nanohydroxyapite (NV_2O_5 and NHAP).

The catalyst (NV_2O_5 and NHAP) was added in concentrations (100, 500, and 1000) ppm to formaldehyde solution at temperatures (25, 50, and 75)°C. The reaction rate increases with increasing the temperature and concentration, where the highest rate of reaction speed was (112.55×10^{-3}) mg / L.min at the concentration of 1000 ppm and a temperature of 75 °C. Table 4. shows the values of the reaction speed rate of (NV_2O_5 and NHAP) with formaldehyde according to the concentrations and temperatures. Increasing the concentration of NV_2O_5 and NHAP, V_2O_5, and NV_2O_5 resulted in a noticeable increase in the reaction speed compared without their presence, which is due to a decrease in the activation energy. Both catalyst concentration and temperature are related to the increase of the reaction speed due to moving the molecules faster and increasing the effective collisions [17,18]. NV_2O_5 demonstrated higher efficacy as a catalyst compared to the examined catalyst.

3.3.4.Activation Energy

Activation energy E_a is a measure of how effective additional energy is in a reaction to accelerate the reaction kinetics, representing it using the Arrhenius equation[19]:

$$K = A \ e^{\frac{-Ea}{RT}} \ \dots\dots (2)$$

where R represents the general constant of gases with value ($R = 8.314 \ J/mol \ K$), K represents the reaction speed constant, A is the Aarhus constant, and T is the temperature.

3.3.5.Nano vanadium oxide (NV2O5)

Fig.8 shows the activation energy behaviour of the (NV_2O_5, V_2O_5, NV_2O_5 and NHAP) and formaldehyde solution reaction at different temperatures and concentrations. The activation energy is decreased for the reaction with increasing the catalyst concentration. Table. 5 shows the values of the activation energy using the prepared catalysts. Catalysts (NV_2O_5, V_2O_5, NV_2O_5 and NHAP) for the different concentrations used in this research. The activation energy of the reactions decreased

with increasing the concentration of the catalyst compared with the interaction without the catalysts, as the principal work of catalysts is to increase the reaction speed due to a decrease in the activation energy[20,21].
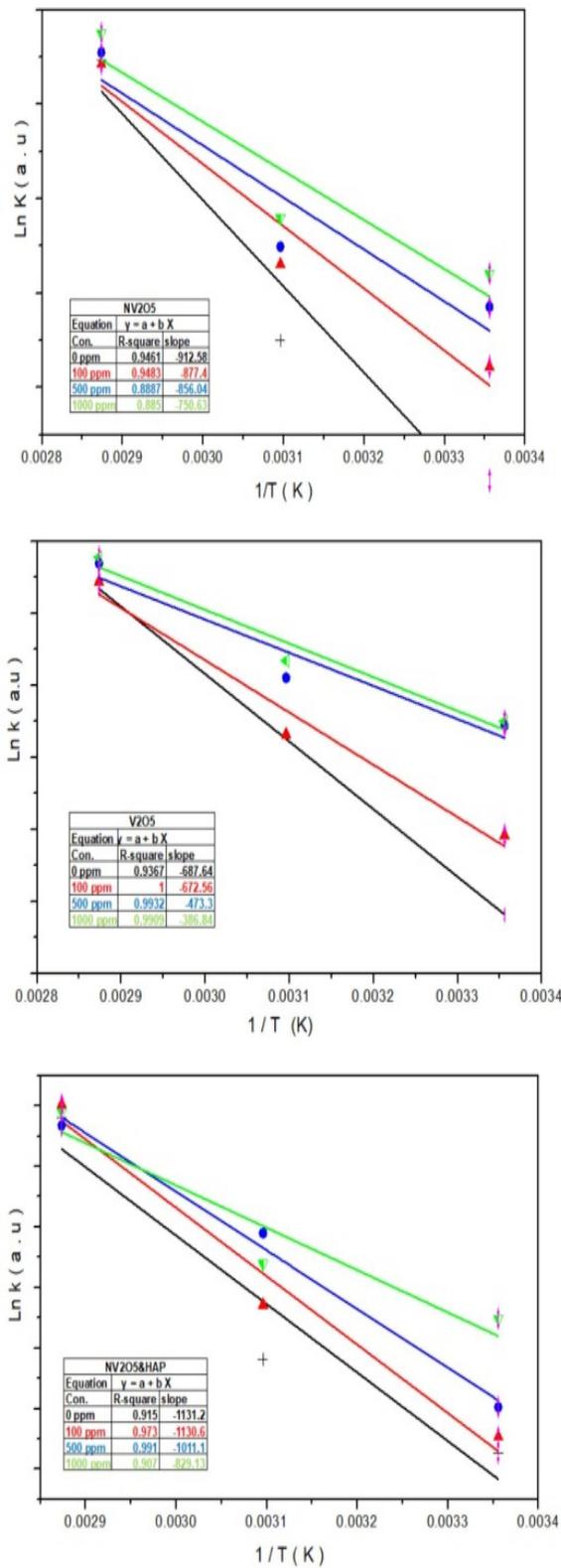
Fig.8: The relationship between inverted absolute
temperature and Ln K using

Table 1: X-ray diffraction parameters of the prepared NV2O5 powder.

No.	Exp.2θ	St. 2θ	h k l	FWHM	Crys. Size(Å)	Chem. formula	Compound name	Crys. system	Card No.
1	11.259	11.318	0 0 1	0.2156	369.68	$H_3O_9V_3$	Hydrogen Trivanadate Hydrate	Monoclinic	98-015-9389
2	15.398	15.362	2 0 0	0.2306	244.89	$H_xV_2O_5$	Hydrogen vanadium bronze	Orthorhombic	00-045-0429
3	17.759	17.844	1 0 0	0.2398	879.67	$H_3O_9V_3$	Hydrogen Trivanadate Hydrate	Monoclinic	98-015-9389
4	22.657	22.702	1 1 $\bar{1}$	0.2504	246.84	$H_3O_9V_3$	Hydrogen Trivanadate Hydrate	Monoclinic	98-015-9389
5	25.049	24.993	0 0 1	0.2216	275.39	V_2O_5	Vanadium Oxide	Orthorhombic	00-053-0538
6	27.733	27.758	1 2 0	0.2185	311.42	V_2O_5	Vanadium Oxide	Orthorhombic	00-053-0538
7	30.76	30.72	2 1 0	0.1562	418.19	V_2O_5	Vanadium Oxide - Gamma	Orthorhombic	98-008-0594
8	40.705	40.71	0 2 $\bar{3}$	0.2077	286.18	$H_3O_9V_3$	Hydrogen Trivanadate Hydrate	Monoclinic	98-015-9389
9	44.734	44.782	1 3 2	0.2455	290.00	$H_3O_9V_3$	Hydrogen Trivanadate Hydrate	Monoclinic	98-015-9389
10	65.28	65.331	3 3 $\bar{1}$	0.1794	1043.79	$H_3O_9V_3$	Hydrogen Trivanadate Hydrate	Monoclinic	98-015-9389

Table 2: X-ray diffraction parameters of the mixture of vanadium oxide and prepared nano-hydroxyapatite.

No.	Ex. 2θ	St. 2θ	h k l	FWHM	Cryst. Size(Å)	Chem. formula	Compound name	Crys. system	Card No.
1	16.788	16.842	1 0 1	1.193	879.19	$(Ca)_{10}(PO_4)_6(OH)_2$	Apatite	Hexagonal	00-055-0592
2	22.159	22.159	1 1 1	1.085	28440.30	$Ca_5(VO_4)_3OH$	Calcium Vanadium Oxide Hydroxide	Hexagonal	00-054-0770
3	25.796	25.879	0 0 2	0.449	168.14	$Ca_5(PO_4)_3(OH)$	Hydroxylapatite, syn	Hexagonal	00-009-0432
4	28.068	28.126	1 0 2	0.268	890.59	$(Ca)_{10}(PO_4)_6(OH)_2$	Apatite	Hexagonal	00-055-0592
5	31.676	31.773	2 1 1	0.554	134.46	$(Ca)_{10}(PO_4)_6(OH)_2$	Apatite	Hexagonal	00-055-0592
6	32.896	32.902	3 0 0	0.502	150.69	$(Ca)_{10}(PO_4)_6(OH)_2$	Apatite	Hexagonal	00-055-0592
7	33.951	33.929	–	0.353	29181.66	$Ca_3(PO_4)_2 \cdot xH_2O$	Calcium Phosphate Hydrate	Un kwon	00-018-0303
8	39.759	39.818	3 1 0	0.947	67.49	$(Ca)_{10}(PO_4)_6(OH)_2$	Apatite	Hexagonal	00-055-0592
9	46.693	46.711	2 2 2	0.607	127.55	$(Ca)_{10}(PO_4)_6(OH)_2$	Apatite	Hexagonal	00-055-0592
10	49.447	49.468	2 1 3	0.607	128.96	$(Ca)_{10}(PO_4)_6(OH)_2$	Apatite	Hexagonal	00-055-0592
11	53.12	53.19	0 0 4	0.456	134.12	$(Ca)_{10}(PO_4)_6(OH)_2$	Apatite	Hexagonal	00-055-0592
12	63.376	63.445	5 1 0	0.391	32799.95	$Ca_5(PO_4)_3(OH)$	Hydroxylapatite, syn	Hexagonal	00-009-0432

Table 3: Average reaction speed using catalyst (NV_2O_5) and catalyst (V_2O_5)

	Reaction rate							
Temp.	ppm 0		ppm 100		ppm 500		ppm 1000	
	NV_2O_5	V_2O_5	NV_2O_5	V_2O_5	NV_2O5	V_2O_5	NV_2O_5	V_2O_5
25 ºC	48.19	30.69	48.01	31.90	54.12	35.68	64.04	42.28
50 ºC	63.31	34.41	72.50	48.70	85.38	59.84	89.06	81.57
75ºC	86.27	43.41	84.80	54.73	107.53	73.77	113.76	100.01

Table.4 :The reaction rate using the catalyst (NV_2O_5 and NHAP)

	Reaction rate			
Temp.	ppm 0	ppm 100	ppm 500	ppm 1000
25 ºC	34.63	36.26	38.78	46.34
50 ºC	38.99	45.99	57.80	64.73
75ºC	67.77	79.07	84.76	112.55

Table.5 :The activation energy of the reaction using the catalysts (NV_2O_5, V_2O_5, NV_2O_5 and NHAP)

Con. (ppm)	Activation energy		
	NV_2O_5	V_2O_5	NV_2O_5 and NHAP
0	7.587	5.717	9.404
100	7.295	5.591	9.399
500	7.117	3.935	8.406
1000	6.241	3.216	6.893

4.CONCLUSION

The reactions of the catalysts (NV_2O_5, V_2O_5, NV_2O_5, and NHAP) for the decomposition of formaldehyde are first-order reactions. The reaction speed and activation energy depend on both the reaction temperature and the catalyst concentration as a function of both temperature and concentration. The reaction velocity is directly proportional while the activation energy decreases accordingly. The decrease in the particle size of the catalysts resulted in an improvement in the speed of formaldehyde decomposition, especially when NV_2O_5 was used as a single catalyst.

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تحلل الفورمالديهايد باستخدام العوامل المساعدة لتراكيب النانوية لأوكسيد الفاناديوم

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[2] جامعة الانبار, كلية العلوم, قسم الفيزياء, العراق

الخلاصة:

تمت دراسة سرعة تحلل الفورمالديهايد باستخدام عوامل المساعدة (جسيمات ثاني أوكسيد الفاناديوم نانوية $(NV_2O_5$)، وأوكسيد نانو الفاناديوم مـع نـانو هيدروكسي أباتيت أكسيد الفاناديوم .تم صنع العوامل لمساعدة بثلاثة تراكيز مختلفة (100 و 500 و 1000) ppm، وفي ثلاث درجات حرارة مختلفة (25 ، 50 ، 75) درجة مئوية. تم استخدام حيود الأشعة السينية (XRD) وصور المجهر الإلكتروني النافذ (TEM) من أجل دراسـة الخصائص التركيبية والهندسية للمحفزات المحضرة، والتي تبين انها مواد ذات بنية نانوية. أدت الزيادة في تركيز المحفزات ودرجة الحرارة إلى زيادة معدل سرعة التفاعلات الكيميائية. ومـع ذلك، فقد خفضت طاقة التنشيط، والتي تم حسابها باستخدام معادلـة Arrhenius. أظهر المحفز أوكسيد نانو الفاناديوم معدل تفاعل أفضل مقارنة بالمحفزات المحضرة الأخرى) للمحفزات أوكسيد الفاناديوم نانوية ونانو هيدروكسي أباتيت أكسيد الفاناديوم على التوالي.

الكلمات المفتاحية: فورمالدهايد ، العوامل المساعدة ، هيدروكسيباتيت ، أكسيد أكسيد الفاناديوم النانوي، التحلل.