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Preparation of Catalyst ZnO-Cd_2O_3 and its Use in Thermal Oxidation of p-xylene in Vapor Phase

Doaa Hamed Salih* and Mousa Umran Kadhum

*College of Education for Girls, University of Kufa**, IRAQ**

Email: doaa.alasady@yahoo.com, musaaldaghr@uokufa.edu.iq

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ABSTRACT

Catalyst was prepared from a mixture of ZnO- Cd_2O_3 by using impregnation Method. Different ratios (2:0, 1.5:0.5, 1:1, 0.5:1.5, 0:2) and supported on granulated kaolin clay with different sizes. The thermal vacuum evaporation technique was at 75°C .Characteristics of the catalyst was studied by using X-ray diffraction (XRD) and some of the physical properties such as surface area , porosity, pore size (Mesh No.) and density. Also Studied the activity of prepared catalyst by thermal oxidation for para-xylene at (315˚c).

Keywords: Thermal Oxidation, p-xylene, Catalysts, kaolin clay.

INTRODUCTION

Catalysts are the most important disciplines of technology in the chemical industry, which has been producing more than 85% of chemical products using the Catalysts. However, its importance is not limited to the chemical industry, it also plays a pivotal role during the processing of raw materials in the refineries, and during the production of energy, such as cells fuel and batteries, as well as in terms of the protection of the environment [1,2], and achieved cofactors developed rapidly in recent years and can be seen in many new applications to it, and cofactors are considered key to the success and development of many new processes in various fields in the industry [3]. Used Catalysts in all sectors of the chemical, which is used in the manufacture of nitric acid, sulfuric acid, ammonia and methanol, as well as in refining , in petro chemistry, in the automotive industry to reduce pollution in the removal of NO, CO and hydrocarbon emissions and reduce consumer waste to prevent environmental pollution [4]. Catalysts are considered key to the success and development of many new processes in various fields in the industry and most Catalysts are in different phases such as solids, liquids and gases, and the interactions of factors assistant is a cyclical process, where the reactants form a complex with a Catalysts, which opens the way for the transformation of reactants to products [5], where it offers a way to change the proportions of the chemical bonds formed and that broken, which can determine the required output which more than unwanted [6]. Cofactors to avoid the use of many of the reagents and solvents, toxic and hazardous and also can reduce a lot of byproducts unwanted [7]. The nature of the catalyst used in industry for cofactor used for laboratory purposes. Factor assistant user laboratory features usually surface area is small and effectively help small compared to his counterpart used in the industry, and is influenced by cofactor laboratory quickly

impurities which works to reduce and reduce laborers assistant while the effect of impurities on the cofactor industrial lower unit also vary the way different method of preparation factor assistant according to use [8]. Thus been defined cofactors as tool to perform chemical reactions in a specific manner with less consumption possible for materials and energy [9], or can be defined are substances that speed up chemical reaction through the formation of bonds with the molecules of the reactants by allowing them (enablers) to interact and configure Output and then separated from the catalyst without any change so that it is available for the next interaction, and interaction can be described as an event cofactor League where co-interaction and back to its original form at the end of the session [10]. Fig 1 can describe the role of catalyst

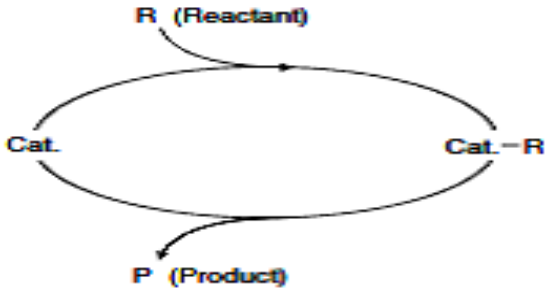


Fig. 1 : The role of catalyst

Classification of catalysts: Catalysts are classified as fitted to two types

1. Metal catalyst : Be in different forms was being tape or wire or metal of the board of thin metallic membrane being deposited on the surface of glass or metal[11,12]. It may be a cofactor metal Condition stuck or colloidal has used Vlzan or more Bhaoh alloy as an adjunct to this is the platinum catalyst successful oxidation of SO_2 is used effectively for the hydrogenation of hydrocarbons, as well as using metal oxides which are semiconductors that works to stimulate the same reaction, but higher temperatures.

2.Compound catalyst : The catalyst composite consists of a single compound or complex numbers or the number of vehicles, but the commonly includes two compounds may be one of the two compounds the amount of small amounts. However, this amount is one of the few compounds useful in increasing and improving the verb worker assistant compound[13].

MATERIALS AND METHODS

Chemicals: Table 1 shows all chemicals been used with their supplier.

Table 1. Chemicals

No.	Chemicals	Purity%	MANUFACTURER
1	Zinc Nitrate , $Zn(NO_3)_2.6H_2O$	98.5	Merch
2	Cadmium Nitrate, $Cd(NO_3)_3.4H_2O$	99	Aldrich
3	para-xylene , C_8H_{10}	98	Merch

Apparatus:

Table 2. List of instruments used in this project.

NO.	INSTRUMENT	MANUFACTURER
1	X-Ray Diffraction	XRD-7000 Shemadzu
2	Surface Area (BET)	HORIBA SA-9600 U.S.A
3	Fourier – Transform ,FTIR	Fourier-Transform 8400S Shemadzu
4	Furnace	Muffle Furnace Size-Two Gallenkamp
5	Oven	Oven Bs Size Two England Gallenkamp

Photo reactor and Procedure: Experiments were carried out in glass photochemical reactor. The cylindrical annular – type reactor consisted of two parts. The first part was an outside thimble, Running water was passed through the thimble to cool the reaction solution. Owing to the continues cooling, the temperature of the reaction solution was maintained of room temperature. The second part was an inside thimble and the reaction solution volume (100 mL) was put in the reaction chamber [14]. Schematic diagram of photochemical reaction as shown in figure 2.

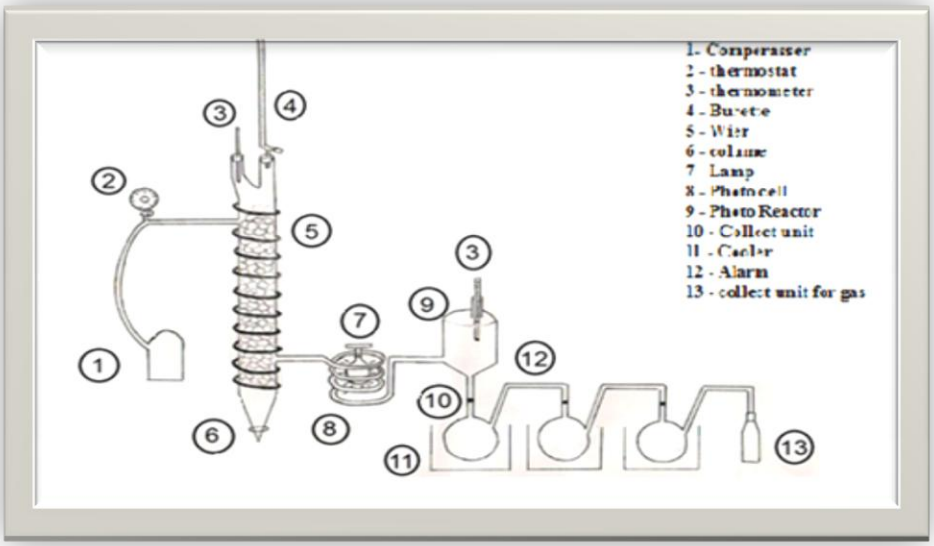


Fig. 2 : Main parts of the System designed for thermal oxidation and optical Preparation of Catalyst

Preparation of Catalyst: A kaolin clay were prepared by mixing quantities of water to form a paste and then it was forming a slurry of different sizes with diameters ranging between (4-2) Micro, and then burned the stone oven to 900^{o} C. Attended the catalyst by mixing zinc nitrate with distilled water as well as the mixing of cadmium nitrate with distilled water, was blending proportions of different size of both solutions prepared in a manner impregnation method). Added sizes of both solutions prepared to the amount of the carrier made from clays A kaolin in each lineage, and are put the mixture in each time to a fumigation vacuum Rotary evaporator to expel the solvent degree of 75°C and then dry cofactor record oven drying degree of 60°C, then were burned pregnant Charge solvents nitrates degree 635°C to turn nitrates to corresponding oxides, were obtained volumetric ratios and the results listed in table 3.

Table 3. List of prepared mixture of ZnO- Cd_2O_3

No.	Volume of zinc nitrate	Volume of Cadmium nitrate	Volume Ratio of mixture
1	2.0	0.0	0.0 : 2.0
2	1.5	0.5	0.5 : 1.5
3	1.0	1.0	1.0 : 1.0
4	0.5	1.5	1.5 : 0.5
5	0.0	2.0	2.0 : 0.0

RESULTS AND DISCUSSION

Structural Characterization: The naked A kaolin clay and prepared mixed Catalyst ZnO- Cd_2O_3 were characterized by

XRD Spectrum:

1. In this technique (XRD) diffraction, we can study A kaolin clay alone as shown in figure 3.
2. In this technique (XRD) diffraction, we can study Zinc oxide with A kaolin clays As shown in figure 4.
3. In this technique (XRD) diffraction, we can study Cadmium oxide with A kaolin clays As show in figure 5.
4. In this technique (XRD) diffraction, we can study Zinc Oxide, Cadmium oxide with A kaolin clays as shown in figure 6.

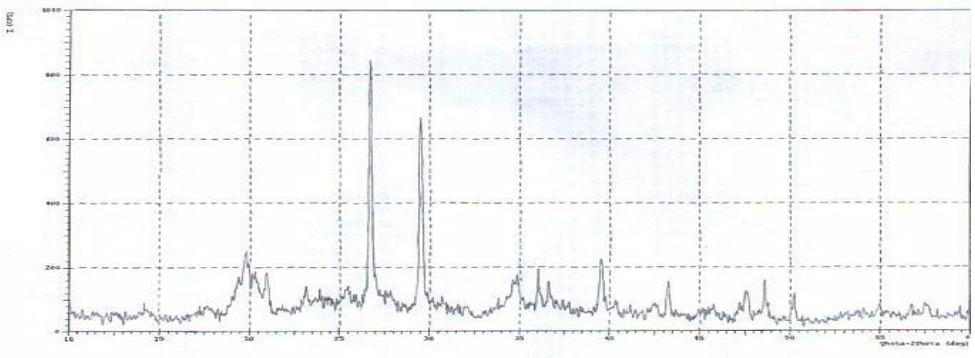
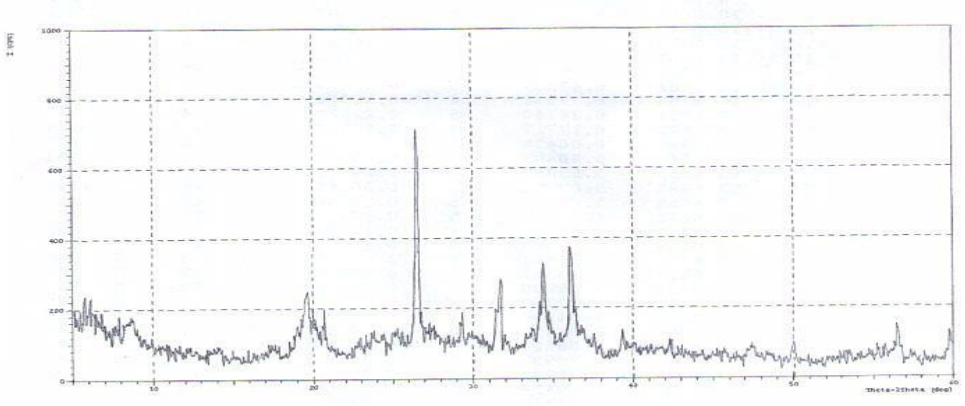


Fig. 3: XRD diffraction spectrum of A kaolin clay

From fig.3, different peaks (2Ɵ) appear in spectrum represent naked A kaolin clay, as show in table 4.

Table. 4 : The value of diffraction angle (2θ) and inter planer spacing (d) for A kaolin clay.

2θ(deg)	**d / Å**
19.8300	4.47362
20.3428	4.36200
26.7453	3.33054
29.8622	2.98963
34.8880	2.56961
36.0790	2.48747
39.7840	2.26394



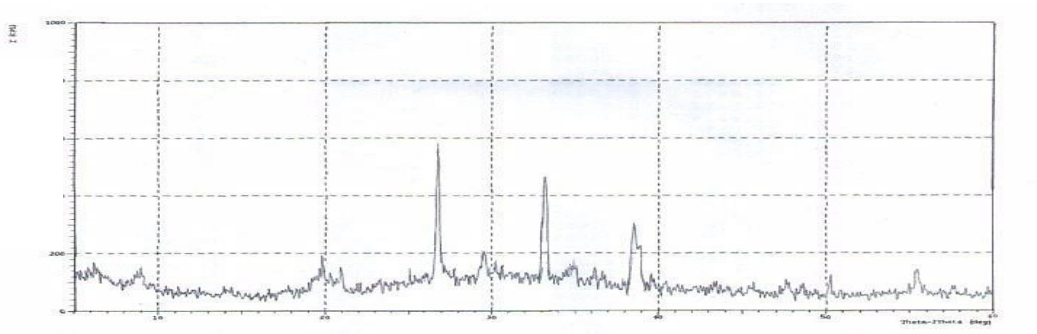
2θ(deg)

Fig. 4 : XRD diffraction spectrum of Zinc oxide with A kaolin clays

From fig.4, different peaks (2Θ) appear in spectrum represent Zinc oxide with A kaolin clays, as show in Table 5.

Table 5: The value of diffraction angle (2θ) and inter planer spacing (d) for Zinc oxide with A kaolin clay .

2θ(deg)	**d / Å**
31.8474	2.80766
34.4869	2.59857
36.1515	2.48264



2θ(deg)

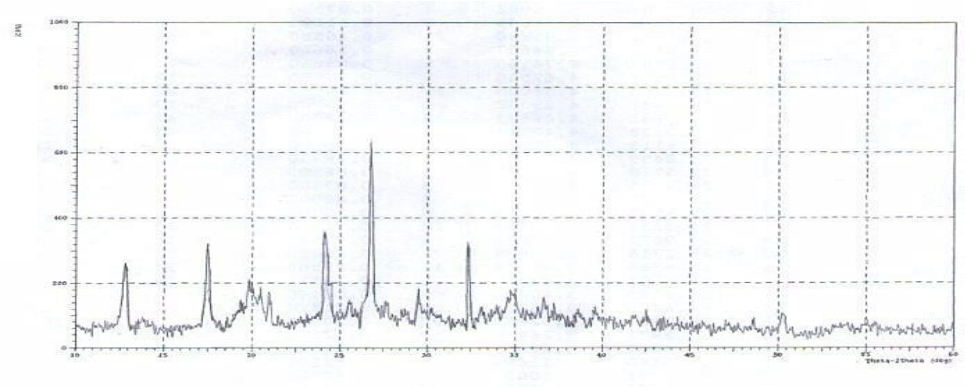
Fig.5 : XRD diffraction spectrum of Cadmium oxide with A kaolin clays

From fig. 5, different peaks (2Θ) appear in spectrum represent Cadmium oxide with A kaolin clays, as show in table 6.

Table.6: The value of diffraction angle (2θ) and inter planer spacing (d) for Cadmium oxide with A kaolin clay**.**

2θ(deg)	d / Å
33.1943	2.69674
38.3958	2.27591

From fig. 6, different peaks (2Θ) appear in spectrum represent Zinc Oxide, Cadmium oxide with A kaolin clays, as given in table 7 .



2θ(deg)

Fig.6 : XRD diffraction spectrum of Zinc Oxide, Cadmium oxide with A kaolin clays

Table.7 : The value of diffraction angle (2θ) and inter planer spacing (d) for Zinc Oxide, Cadmium oxide with A kaolin clays.

2θ(deg)	**d / Å**
12.8478	6.88484
17.5190	5.05820
24.3131	3.65793
32.5411	2.70820

Study the Physical properties of catalyst: A study of some physical properties of the mix ZnO-Cd_2O_3 as a ratio of 1:1 (50%), a clear divergence from the study of the physical properties of the other ratio.

Surface area of prepared catalyst: Method is based on the usual to measure the surface area of worker Assistant percentage volumetric (50%) on the physical adsorption of nitrogen gas flowing on the surface of the steel worker Assistant subject in the cell-shaped (U) in the measuring device surface area (HORIBA) cell placed in a bath containing nitrogen liquid cooling cell model for the occurrence of adsorption of gas on the surface of the catalyst, are measuring the amount of nitrogen adsorbed at equilibrium conditions at the boiling point (190 K) and pressures of different nitrogen less than atmospheric pressure results were measuring the surface area of the worker aid record percentage volumetric (50%) and found equal to the surface area (51.8377 m^2 g^{-1}).

Porosity of Prepared Catalyst: Know porosity as the amount or the number of voids in the catalyst, and the estimated porosity using the method of marinating is a way to drain catalyst then weighed ,Wt_1 and then drown the catalyst in a liquid (oil) and boil for 5 min and then cooled Allbeckr which contains a catalyst and oil in a water bath cool for 5 min then transported beads cofactor to cloth damage out to remove excess oil and then weighed represents the weight, Wt_2. attached beads cofactor in the oil by placing them in buckle is connected to the arm of a sensitive balance and weighed in terms of a weight, Wt3 and porosity is calculated according to the following relationship :

$$P = (Wt_2-Wt_1/ Wt_2-Wt_3) \times 100 \text{ ..... } (1)$$

After application of the law, above results were obtained presented in table 8.

Table.8: The porosity at different percentage of volume.

No.	Volume Percentage%	Porosity of mixed Zinc oxide and Cadmium Oxide
1	0	36.283
2	25	49.242
3	50	71.200
4	75	55.555
5	100	42.574

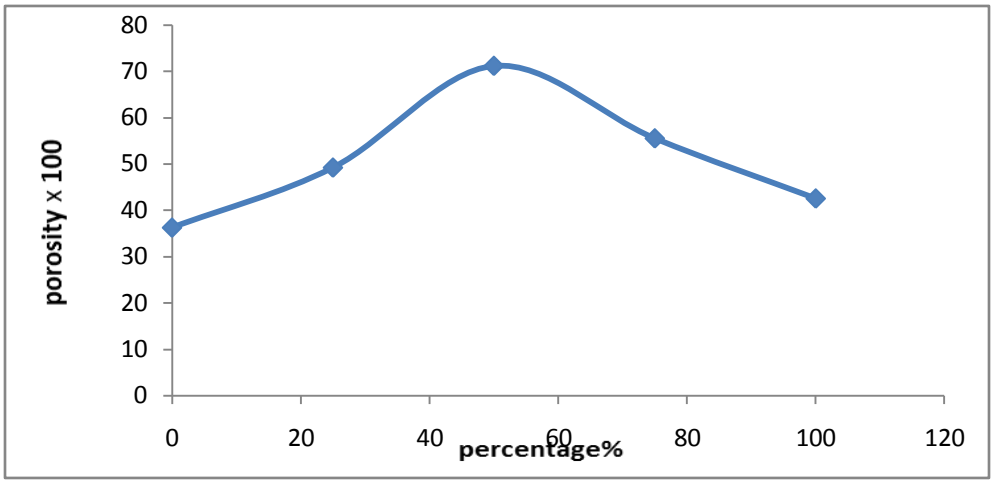


Fig. 7: The relation between porosity at different percentage of volume.

The measured porosity of the factors helping the prepared percentages are different, where it was observed that increase the value of the porosity, increase volumetric percentages to reach the highest value at the percentage volumetric 1:1 (50%) after decreasing. The reason for this removal of water interdependence between oxides and impregnated record and the holes and pores as a result of blowing gas, carbon dioxide. The holes are the result of random association between Alaoxadan min because of the difference between the phases of the system to a combination of crystalline oxides.

Pore Size of Prepared Catalyst: Defined as the space between the atoms, which facilitates the process of adsorption of reactive material, and measured the pore size of the catalyst using drenching method, were obtained on the pore size of the catalyst record using the relationship:

$$\mathbf{V = (Wt_2 - Wt_1 / Wt_1) \times \rho .... (2)}$$

Where ρ: density of oil, Wt_1 : the weight of a grain of cofactor which is dry,Wt_2 : the weight of a grain of catalyst after being boiled in oil

After application of the law above results were obtained in the following table (9) :

Table.9: The Pore size at different percentage of volume.

No.	Volume Percentage%	Pore size of mixed Zinc oxide and Cadmium Oxide
1	0	0.096
2	25	0.1344
3	50	0.2002
4	75	0.1861
5	100	0.1051

Note increase the value of the pore volume increase with percentages volumetric to be up to the highest value at the percentage volumetric 1:1 (50%), then decreased and the reason that the pore size between the minutes be less as possible when they are oxides single reason for this is due to the stacking and the convergence of the min asymmetric be larger, while the pool gets random minutes between various oxides as a result of the fact that the stacking and the convergence of different minutes be less and this leads to increase the pore size between different min.

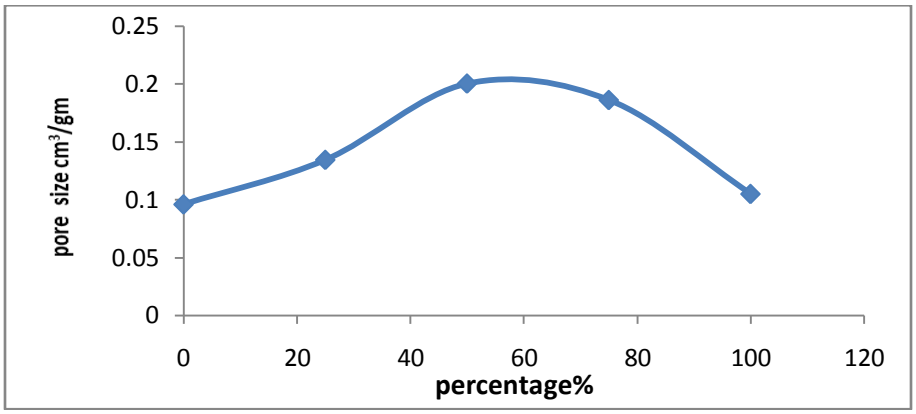


Fig.8: The relation between pore size at different percentage of volume.

Density of Prepared Catalyst: Known as the mass density and unit volumes, the density was measured catalyst percentage volumetric different way drenching and the application of the results that have been obtained using the relationship:

$$\rho D = Wt_1 / Wt_2\text{-}Wt_3 \quad \ldots\ldots\ldots (3)$$

After application of the law, above results obtained were presented in table 10.

Table. 10 : The Density at different percentage of volume .

No.	Volume Percentage%	Density of mixed Zinc oxide and Cadmium Oxide
1	0	3.0584
2	25	2.9659
3	50	2.4176
4	75	2.88
5	100	3.2792

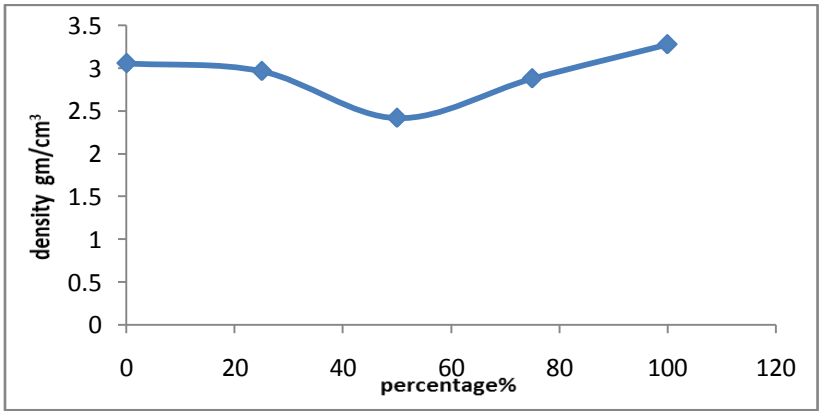


Fig.9: The relation between Density at different percentage of volume.

Where we note a decrease in the density of the catalyst, which was prepared percentages of different size to reach the lowest value at a 1:1 volumetric percentage (50%) and then go back to growing, where due to the random arrangement minutes mix between what could be the biggest .

The study of factors affecting the efficiency of the catalyst : Been studying the factors influencing the efficiency of the catalyst by thermal oxidation and the duration of heating 3 h in a row and was the air

pressure and the speed of its neighbors within the reactor 14psi and 8.5 cm^3 sec^{-1} respectively. The reaction product was diagnosed by a device measuring the infrared.

The effect of the volumetric proportions of the oxidation factor Assistant p - xylene thermally at a temperature of 315 °C and the air: Has been conducting a series of thermal reactions, using zinc oxide - cadmium oxide loaded on Alcaaalin Clay and proportions [2: 0, 1.5: 0.5, 1:1, 0.5:1.5, 0: 2] was the use of the following relationships to calculate the amount resulting from the oxidation of the para - xylene heat and air presence, namely:

$$\text{Percentage of product \%} = \frac{\text{number of product moles of acid}}{\text{number of moles of reactant}} \times 100 \quad \text{.....(4)}$$

$$\text{Percentage of conversion \%} = \frac{\text{number of conversion moles}}{\text{number of moles of reactant}} \times 100 \quad \text{... (5)}$$

The results are shown in table11.

Table 11: The Product percentage at different percentage of volume.

No.	Percent volume	Time/ hour	Product percentage	Percent of conversion
1	0	4.5	0	0
2	25	4.5	23.78	12.994
3	50	4.5	34.628	15.998
4	75	4.5	28.979	13.683
5	100	4.5	0	0

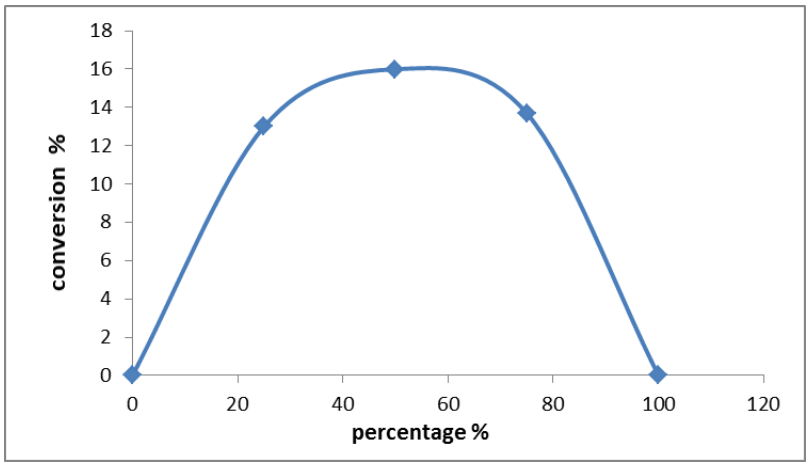


Fig. 10: The relation between percentage conversion of p - xylene at different percentage of volume.

It was found that the highest amount of output with less percentage of transformation of a substance p - xylene when the ratio (50%) 1:1 indicating that cofactor record by (50%) 1:1 ratios more efficient than the other, and the reason for this then get higher tangle and Tasr between Alaoxadan where we believe that every minute of zinc oxide are stirring minutes of cadmium oxide

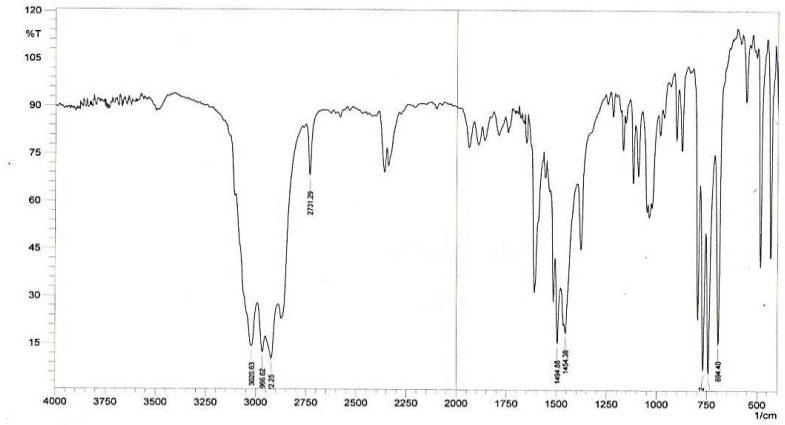


Fig.11: FTIR Spectrum for para - xylene before oxidation

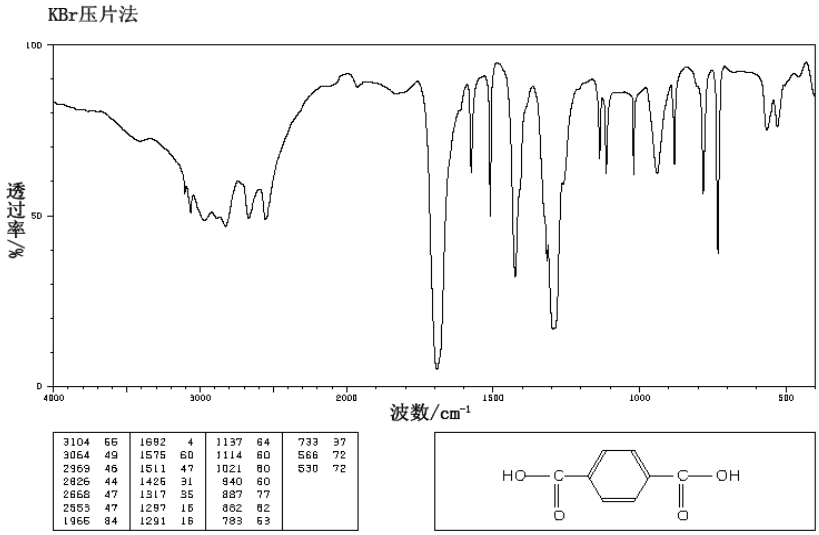


Fig.12: FTIR Spectrum for Pure terphthalic acid

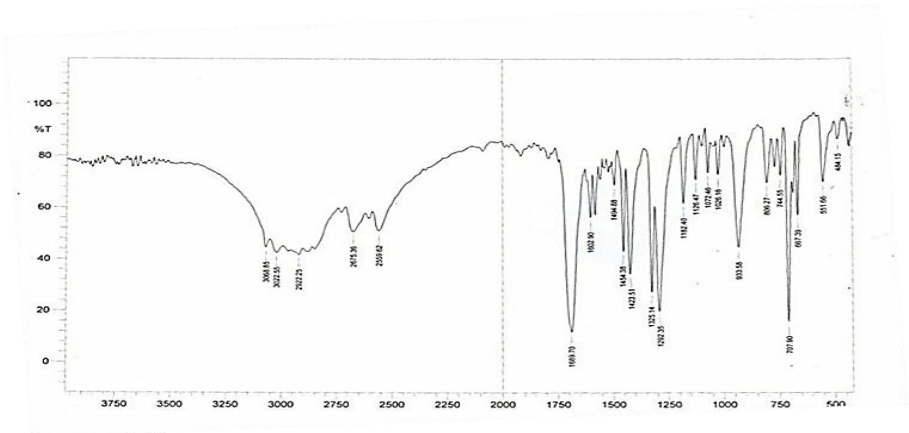


Fig.13: FTIR Spectrum for terephthalic acid obtained from thermal oxidation for para - xylene by using mixed ZnO- Cd_2O_3 (1 :1).

APPLICATIONS

The prepared catalyst is used successfully for thermal oxidation of p-xylene.

CONCLUSIONS

1. The catalyst ZnO- Cd_2O_3 has been prepared using impregnation Method.
2. The physical properties of catalyst ZnO- Cd_2O_3 has been studied such as pore size, porosity and density, the optimum at percentage (1:1).
3. The activity of catalyst ZnO- Cd_2O_3 at (1:1) has been studied using p - xylene.

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