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THE INFLUENCE OF FLOW RATE IN THE ETHERIFICATION OF
DIISOPROPYL ETHER VIA TECHNOLOGY OF AEROSOL NANOCATALYSIS

This paper proposes and describes a new method for the production of Diisopropyl ether from the catalytic dehydration of isopropyl alcohol, using the technology of aerosol nanocatalysis on a vibrating fluidized bed. The catalyst used was NaX, which was later treated with ammonium nitrate, in order to increase its acidity and bridge the OH group, which in turn increased the bronsted acid sites. Parameters such as flowrate, temperature were varied while keeping the mechanical chemical activation frequency of vibration constant in order to understand the relationship between flow rate of reactants into the reactor and the mechanical chemical activation frequency so as to optimize conditions for the synthesis of Diisopropyl ether through catalytic dehydration of isopropyl alcohol.

Keywords: aerosol-nanocatalysis, , vibrating fluidized-bed, flowrate, IPA ,DIPE.,NaX-Zeolite

1. Introduction. The need to eliminate lead-based octane enhancers in gasoline has provided an incentive for development of processes to produce high octane gasolines blended with lower aliphatic alkyl ethers as octane boosters. Supplementary fuels are being vigorously developed in the petroleum refining industry. Lower molecular weight alcohols and ethers such as isopropyl alcohol (IPA), and diisopropyl ether (DIPE) are in the boiling range of gasoline fuels and are known to have a high blending octane number. They are useful octane enhancers. In addition, by-product propene (propylene) from which IPA and DIPE can be made is usually available in a fuels refinery, typically as a C₃+aliphatic stream rich in propene and propane. Feed stocks of particular interest are light hydrocarbon streams rich in propene, which are often accompanied by significant amounts of propane, ethene, ethane and other light gases. The production of ether from secondary alcohols such as isopropanol and light olefins is known. As disclosed in [1], DIPE is produced from IPA and propylene in a series of operations employing a strongly acidic cation exchange resin as catalyst. Recently, processes for the hydration of olefins to provide alcohols and ethers using zeolite catalyst have been disclosed by [2, 3, 4, and 5]. These applications are incorporated herein in their entirety by reference.

Due to this, the dehydration of secondary alcohols e.g isopropanol is a very interesting process, because the activity and selectivity of dehydration reactions are controlled by texture, acidity and electronic properties of the catalyst [6]. The conversion of isopropyl alcohol has been related to the presence of both acid and basic sites on the surface of the catalysts, these sites are mostly found in zeolite catalyst simultaneously. In the broad outline, isopropyl alcohol dehydrates to propene and diisopropyl ether over acid catalysts and dehydrogenates to acetone over basic catalysts [7, 8].

Diisopropyl ether which is a byproduct of dehydration of isopropanol has been found to be a useful oxygenated additive to motor fuels and as antiknock components in gasoline, because it does not have the following drawbacks like easy diffusion in the ground water, slow biological breakdown and unpleasant odor currently experienced in the use of MTBE (tert methyl butyl ether) and the world wide food shortage that might be experienced if ETBE (tert ethyl butyl ether) is continuously produced from corn and other food sources. Diisopropyl ether (DIPE) can be a potential candidate to substitute for MTBE. It has the advantage to increase the octane number and to have a lower vapour pressure. Various works proposed the synthesis of the DIPE from propene and water in a two-stage process, from a feed of acetone and hydrogen in a two-stage process [9] or onestep process [10] and from acetone feedstock [11]. DIPE can also BE synthesised either from a feed of propylene and water [12] , from a feed of propene and isopropanol over an acidic ion exchange resin like Amberlyst15 [13] or from a condensation of isopropanol on heteropoly acid catalyst. It is well documented that the synthesis of isopropanol to DIPE, depends on the efficiency of the catalyst (highly acidic catalyst), the operating parameters rather than the rate of dehydration of IPA or the reaction of IPA with propylene. It is also stated that pressure in which reactions are catalysed does not have a large effect on the rate of DIPE production, in as much as the temperature used. The most important factor in DIPE synthesis from IPA ,is that the liquid phase has to be maintained [14]. Common catalysts that have been used in DIPE synthesis are Montmorillonite clay catalyst, Ion exchange resins, Heteropoly acid catalysts and Zeolites , the latter has been considered to be a better catalyst because it can withstand high temperatures.[15]. The synthesis of DIPE does not have a direct dependence on pressure using a vibrating fluidized bed reactor under aerosol nanocatalysis technology, as it operates under atmospheric pressure, and the vibration of the reactor induces a liquid phase within the reactor, which enables the synthesis to take place., thereby eliminating the need for outside source of pressure. The foundations of AnC were developed by Soviet scientists under the leadership of Glikin M. in the Severodonetsk branch of the State Institute of Nitrogen Industry [16]. At the heart of AnC technology are mechanochemical effects arising from the constant mechanical action of the catalytically active substance. The catalyst system in AnC technology is a moving dispersing material. During the continuous movement of the dispersing material, there is a constant mechanochemical activation of the catalyst that occurs, which leads to a change in the thermodynamic potentials of the activated substance, creating surface defects, and eventually leading to the grinding up to the nanoscale state [17].

The main goal of the study was to:

- Evaluate the main parameters of the catalytic dehydration of isopropyl under the conditions of aerosol nanocatalysis technology in a vibrating fluidized bed reactor by keeping the mechanical activation frequency constant and varying the temperature and flow rate.
- Implementation of the basic principles of AnC technology in a vibrating fluidized bed reactor, by conducting experiments on the catalytic dehydration of isopropyl alcohol;
- Proof of the course of catalytic processes in a laboratory installation,
- Detailed analysis of experimental data and selection of the most optimal variant of process parameters

2. Analysis of the current level of research in the field of AnC. The technology of Aerosol nanocatalysis is founded upon the mechanism of mechanochemical activation, which is used to improve the performance of the catalyst [17,19]. During mechanical interaction, the temperature rises at the points of contact and relaxation of the stress field [18, 19]. As a result of the moving solids, most of the work which would have been done against frictional forces manifests itself in the form of heat. In the surface layer, the temperature can instantaneously increase to 1300°K. The bulk of the substance at this time remains cold. But pulses of high energies can lead to the sintering of nanoparticles. The time of existence of such temperature flares on an area of 10^{-7} - 10^{-9} m² is of the order of 10^{-4} s. Emerging freshly formed surfaces on the catalyst carry an electric charge. As a result, the destruction of reagents is likely on the surface layer. Under optimal conditions, defects are formed on the surface of the crystallites and the electronic properties of the surface change, which leads to an increase in the catalytic activity of the substances, the increase in catalytic activity is believed to be associated with crystal lattice distortions, rather than with an increase in surface area and this provides, new opportunities for controlling the kinetics of processes [17].

The implementation of catalytic processes in the conditions of AnC technology (without carrier) leads to a decrease in the amount of catalytically active substance (up to 10^6 times), and also removes mechanical and thermal limitations associated with the use of a carrier [20]. Under the conditions of this technology, it is possible to reduce the temperature range of the course of destructive transformations and increase the conversion of raw materials.

At the next stage of the development of AnC technology, it was suggested to carry out studies in installations with a vibro-impact or vibrating layer, where the mechanochemical activation of the catalyst is carried out by vertical reciprocal movements of the laboratory reactor and the catalytic system inside it [26]. The process was called Aerosol nanoCatalysis with Vibrating Bed (AnCVB). The capacity of the reactor with the vibro-impact layer turned out to be higher than that of the reactor with a fluidized bed, in addition, new opportunities for controlling the kinetics of processes [21].

3. Experimental part.

AnC catalyst system consists of a catalytically active material powder with initial particle size of 200 microns and its dispersing solid material (mm) with a size of 1.0-1.2 mm. Both types of particles in the reactor are in constant motion. Such conditions allow the synthesis of nanoparticles of different size by mechanical action on the raw material dispersing catalyst particles [22]. In previous studies [23] with the aid of foreign scientists we were able to identify directly catalyst particle sizes during the reaction: 8-100 nm [24].

Previous work established that before the start of the experiment there should be starting materials for preparation of the catalyst system. A specific feature of AnC is the adhesion (type sorption) of the catalyst on the surface of the dispersing material [22]. It is necessary to ensure maximum adsorption of the catalyst particles in a reactor under conditions of vibration. This allows determining the amount of catalyst in the gas phase.

Before starting the experiment, the catalyst should be pre-adsorbed onto the surface of the dispersing material. Work is carried out as follows: for the experiment a certain amount of dispersing material (in laboratory experiments, is the glass bead with a diameter of 1.0-1.2 mm), which occupies about 50% of the reactor volume. Dispersing the material in an amount more than necessary is loaded into a glass flask. To this was introduced the catalyst powder that was weighed on an analytical balance to the nearest 0.0001 gm, in an amount exceeding the adsorption capacity of dispersing material. The flask was covered and required a vigorous shaking performed at room temperature for 10 - 15 min. After shaking, the catalyst and the dispersing material are loaded into the vibrating reactor, and the reactor is operated at a vibration frequency to enable the total dispersion and heating of the catalyst on the dispersing material. Then, the catalyst system is discharged from the flask and sieved in order to separate the catalyst which was not adsorbed. The separated catalyst is weighed on an analytical balance. If the mass of the catalyst is less than the initial amount, a new measured catalyst is added to the glass beads in a flask, and then shook vigorously, and later loaded again unto the reactor which vibrates on a non definite MCA frequency for 1 hour. The process is repeated until; there is no change in mass from the unloaded catalyst after heating in the reactor, in comparison with the 1st previously weighted catalyst after shaking in a flask. [24]

Previous studies have shown that in the absence of a catalyst in gas phase, as when it is excess, the reaction rate is minimal [24]. The catalyst powder is weighed and added to the reactor so as to create a constant concentration in the reaction volume. The reactor was checked for leaks before conduction of experiment [24].

Description of experimental setup: The process of synthesizing DIPE from catalytic dehydration of isopropanol using aerosol nanocatalysis technology was conducted in a laboratory setting, shown in Fig. 1 [20, 21].

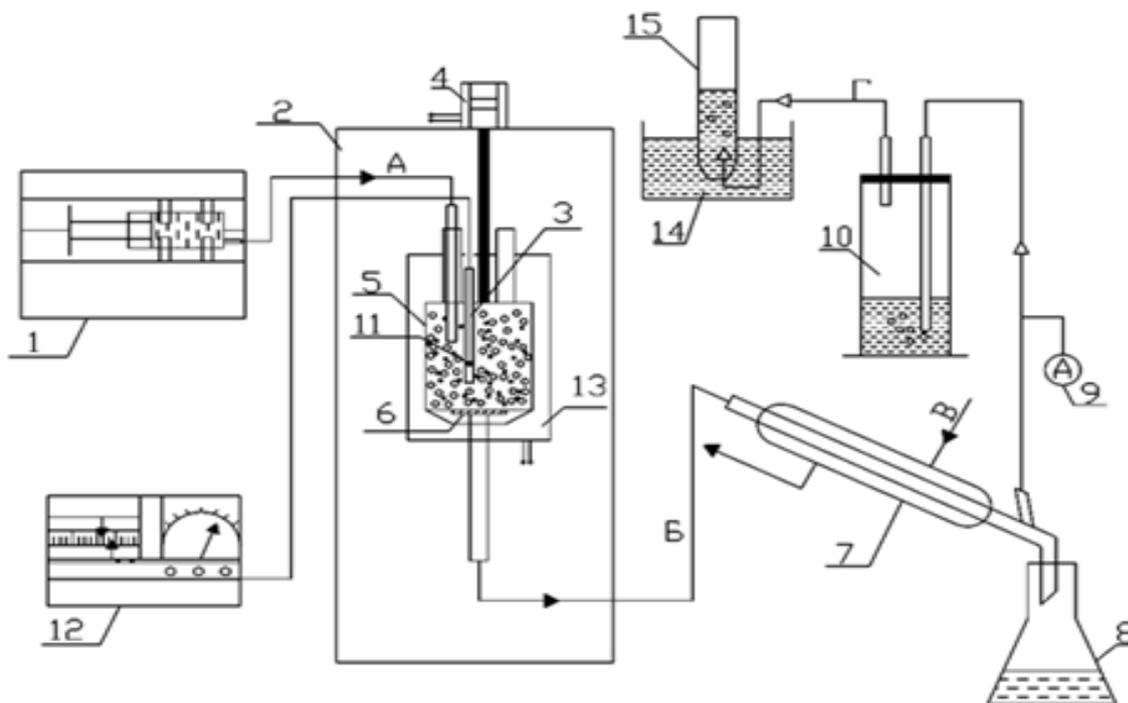


Fig.1. Schematic diagram of the pilot unit

(1) - Syringe batcher; (2) - Thermal cabinet; (3) - Pocket of thermocouple; (4) - Vibrating device; (5) - Reactor; (6) - Metal-cloth filter; (7) - Water condenser; (8) - Receiver of liquid fraction; (9) - Sampling point; (10) - Gas washer; (11) - Thermocouple; (12) - Regulator for oscillation frequency and temperature; (13) - Heater; (14) - Water container; (15) - Gas receiver.

Before each reaction, the catalyst was preheated in situ for 2 hours at 150°C-300°C (heating rate of 5°C/min) under nitrogen steam with a flow of 2 L/h, after which 20 ml of isopropanol was fed into the reactor (5) by a syringe-batcher (1), which is located in the thermal cabinet (2), and it was then subjected to thermal treatment by heater in the reactor (13). Etherification reactions took place inside the reactor. Experiments were conducted at temperatures: 140°C, 160°C, 180°C, 200°C, 220°C, 240°C, with a variation in the flow rate of the reaction, the reaction rates were as followed 0.1 ml/min, 1.0 ml/min, 1.9 ml/min, and 3.7 ml/min, and at a frequency of 1.8 Hz. The temperature in the reaction zone was measured by a thermocouple (11), and sustained by a regulator (12). In order to keep the catalyst inside the reactor, a metal-cloth filter (6) was placed at the bottom of reactor, the liquid reaction products pass through a water-cooled condenser (7) and were then gathered in a flask for the liquid fraction (8). The etherification products were two fractions – liquid and gas. The non-condensed gases from the cracking process go through the sampling point (9), and were then sent to the gas washer (10) where they bubble through the water layer. The gases were then directed into a gas container (15), and passed through container with water (14). The reactor moves upward and downward in a back-and-forth linear motion with the help of vibrating device (4). The oscillation frequency and temperature were set and controlled by a regulator (12). The liquid phase was subjected to extractive distillation, by using an extractive distillation agent in the form of glycol ether so as to separate the resulting mixture into their individual component. This was achieved by using a rectification column, that contained the products from the reactor, and subjecting it to extractive distillation, the extracting agent was introduced a few plates from the top of the column, to ensure none of the extractive agent is carried over with the component of the highest vapor, this requires that the extractive agent boils 20°C or more higher than the highest boiling component. Isopropyl alcohol, diisopropyl ether and water from 3 binary azeotropes and 1 ternary azeotrope [25].

The content of the gaseous products of the reaction were analytically determined by using calibrated gas chromatographs such as LHM-8, COLOR-500. Propylene (C_3H_6) was determined with an accuracy not less than 0.01 vol. %.

The products gotten from extractive distillation, were analysed with an online gas chromatograph with a 2-m (i.d., 3mm) column of 8% carbowax 1540/chromosorb (W80-100 mesh) and a flame ionization (FID) detector (Delsi ICG 121 MI). The results gotten from the chromatograph indicated the presence of DIPE, IPA and water.

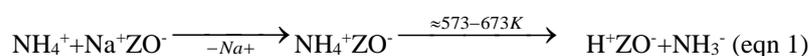
Azeotrope of Isopropyl ether,IPA and water

Table 1

COMPOUNDS	BOILING POINT °C	Azeotrope composition % wt		
WATER	100			
ISOPROPYL ALCOHOL	82.5			
DIISOPROPYL ETHER	69		87.4	
WATER-ISOPROPYL ALCOHOL	80.3	12.6	83.7	
ISOPROPYL ALCOHOL-DIISOPROPYL ETHER	66.2	16.3	95.5	
WATER-DIISOPROPYL ETHER	62.2	4.5	7.3	
WATER-ISOPROPYL ALCOHOL-DIISOPROPYL ETHER	61.6	4.7		88

4. Materials and research results. . The NaX zeolite used in this study was obtained from REAXIM Ukraine. It was in the form of 1.5mm extrudate. The Isopropyl alcohol was also purchased from REAXIM Ukraine.

Catalyst preparation: 10grams of NaX zeolite was first calcined to remove moisture and kept ready for ion exchange. Then the zeolite was treated at 200°C, with excess aqueous ammonium nitrate, the aqueous ammonium nitrate was prepared by dissolving 10 grams of ammonium nitrate in 100 grams of distilled water, and this was heated to 80°C. Thereafter the suspension was filtered. The filtrate had an increased Na content of 0.4% by weight. The filtered zeolite was treated again treated in an ammonium nitrate solution of 10 grams of ammonium nitrate and 100 grams of water, which had also been heated for 2 hours. This was followed by filtration and washing of the zeolite with 180 grams of distilled water. The wet filter cake after washing was dried at 120°C for 4 hours and then calcined at 500°C for 5 hours. The calcinations enabled the release of ammonia. Prior to use, thermally treated samples were stored inside air-tight vials to prevent the absorption of moisture from the atmosphere. Characterization of structure, the size, and morphology of particles were determined by XRD techniques. N₂ adsorption isotherm was measured at 77.5K using a Micromeritics ASAP 2020



The goal of this research was to find out how the speed of reaction and residence time of reactants in the reactor influenced the synthesis of DIPE using the conditions of aerosol nanocatalysis on a vibrating fluidized bed, considering ACnVFB offers the following advantages, that are not common with other etherification reactions like:

- Elimination of intradiffusion reaction stages
- Equal access of the reagents to the catalytically active surface;
- Strength and heat resistance of the catalyst;
- Use of a simple catalytic system;
- Creation of an optimum quasi-structure of the catalyst, controlling its concentration in the reaction zone;
- Mechanical activation of the catalyst in situ with changes in the surface properties of catalysis important for catalysis
- Achievement (in terms of mass) of the aerosol activity of the catalyst which is 10⁴-10⁵ times higher than that of the carriers;
- Possibility of catalyst regeneration by mechanical surface treatment in situ;
- Reducing the amount of catalyst used in the reactor by 10⁵-10⁶ times and bringing it to 4-10 g / m³ of the reaction volume. [27].

Below are the results obtained from synthesis of DIPE using modified H-X zeolite, with variation in temperature and rate of reaction, while keeping the mechanical chemical frequency of vibration 1.8Hz constant.

Table 2

Results obtained from synthesis of DIPE using H-X zeolite, keeping Mechanical Chemical Activation frequency constant, while varying the flow rate and temperature

Flow rate of 0.03ml/min					
T, °C	Liquid product(ml)	% Conversion IPA	Volume of H ₂ O (ml)	% Mass Selectivity DIPE	Volume of DIPE(ml)
140	17.9	0.195	1.3	0	0
160	18.2	0.165	1.2	0	0
180	18.1	0.37	1	64.5	3.9
200	18	0.47	0.8	75.12	5.9
220	18.4	0.52	1.1	79.06	7.5
240	18.3	0.4	0.9	30.717	0.8
Flow rate of 0.1ml/min					
140	18.3	0.15	0.9	0	0
160	18.3	0.175	0.8	0	0
180	18.2	0.405	1.9	53.2	3.4
200	18.3	0.495	1.6	68.45	5.6
220	18.1	0.55	1.8	68.49	6.2
240	18.2	0.45	5	10.514	0.7
Flow rate of 1.0ml/min					
140	18.1	0.19	0.9	0	0
160	18.3	0.17	0.8	0	0
180	18.2	0.31	1.9	50.126	3.1
200	17.9	0.47	1.6	63.198	5.0
220	18.3	0.495	1.8	67.952	5.6
240	18.2	0.46	4	12.218	0.69
Flow rate of 1.9ml/min					
140	18.2	0.15	1	0	0
160	18	0.2	1.5	0	0
180	18.1	0.4	3.2	36.614	2.3
200	17.9	0.345	1.5	48.949	2.6
220	18.3	0.37	1.3	66.755	4.5
240	18.4	0.425	3.9	8.299	0.43
Flow rate of 3.7ml/min					
140	18.1	0.15	1	0	0
160	18.1	0.2	1.6	0	0
180	18.2	0.35	2.5	0	0
200	18.3	0.325	2.8	0	0
220	18.1	0.335	1.9	35.151	1.6
240	18.2	0.35	1.6	3.763	0.1

Based on the following experiments, it was evidenced that flow rate and temperature go in tandem to achieve maximum selectivity to DIPE, the lower the flow rate (high residence time), the higher the synthesis of DIPE, this could be attributed to the fact that a decreased flow rate enables the reactant to have sufficient access to the surface of the catalyst, which in turn increases the activity of the catalyst. Also it was observed that DIPE synthesis using aerosol nanocatalysis technology on a vibrating bed has a definite temperature in which DIPE can be synthesized, and any reaction above this temperature leads to production of propylene, but these two factors must work together, in order to achieve the desired result

Conclusions.

1. In the course of the experiments, the basic principles of AnC technology were realized in a vibrating bed reactor, etherification experiments were conducted on the catalytic dehydration of isopropanol to DIPE.
2. The technique of carrying out the experiments was worked out: the integrity of the installation was ensured as it was immune to leakage and the reproduction of the results was achieved with satisfactory accuracy.
3. The experimental method proves the course of catalytic processes in the reactor. The effect of temperature, residence time of reactant on the yield of the desired product was studied.
4. During the course of experiments, – the ideal flow rate of the reactant into the reactor was 0.03ml/min and the ideal temperature was 220°C, after which the etherification reaction yielded more of propylene.
5. In order to separate the products gotten from the reaction into their distinct components, an extractive agent is needed to enable such separation, and this agent must boil 20°C or higher than the highest boiling component.
6. The vibrating conditions of the fluidized bed enabled the reaction to be maintained in the liquid phase, which is a prerequisite in the synthesis of DIPE from isopropyl alcohol.
7. Strong bronsted acid sites are essential in the synthesis of DIPE from isopropyl alcohol using zeolite catalysts, and these bronsted acid sites can be induced into the catalyst by treating the zeolites with ammonium nitrate, ammonium carbonate (ion exchange) etc.

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В статті запропоновано та розглянуто новий метод виробництва діізопропилового ефіру шляхом його каталітичної дегідратації в умовах технології аерозольного нанокаталізу із віброзрідженим шаром каталітичної системи. В якості каталізатора використовувався цеоліт NaX, який в подальших експериментах було оброблено нітратом амонію для збільшення вмісту –ОН груп та відповідного збільшення Бренстедевських кислотних центрів. Такі параметри, як витрата сировини та температура процесу, змінювалися, зберігаючи частоту механічної хімічної активації постійною, щоб зрозуміти зв'язок поміж швидкістю реакції та частотою механічної хімічної активації, з тим щоб оптимізувати умови для синтезу діізопропилового ефіру шляхом каталітичної дегідратації ізопропилового спирту.

Ключові слова: аерозольний нанокаталіз, віброзріджений шар каталітичної системи, ізопропанол, діізопропиловий ефір, цеоліт NaX.

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