

# Microreactor Technology for Fine Chemicals

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**Abstract:** Using biological reactions, microreactors produce fine chemicals such as perfumes, flavors, and fragrances. The development of biotechnological processes for the production of fine chemicals using micro reaction technology has been driven by environmental regulations, cost competitiveness and public awareness on safety issues. Microreactors offer several advantages over traditional chemical synthesis techniques regarding process efficiency, product purity and shorter reaction times. In addition to these benefits, microreactor technology is also applicable to producing fine chemicals derived from renewable raw materials. The microreactor technology offers a cost-effective alternative to traditional synthesis techniques in terms of product yield and purity, thus opening up new opportunities to develop fine chemicals derived from renewable resources. This paper researches microreactor technology. It describes the concept of the microreactor, its structure and working principle. As microreactor technology is very mature, it has been used in many real industrial production processes. From the viewpoint of application, we summarize and analyze several typical examples. This paper also tries to explore existing problems in the application of microreactor technology and to find out its prospects.

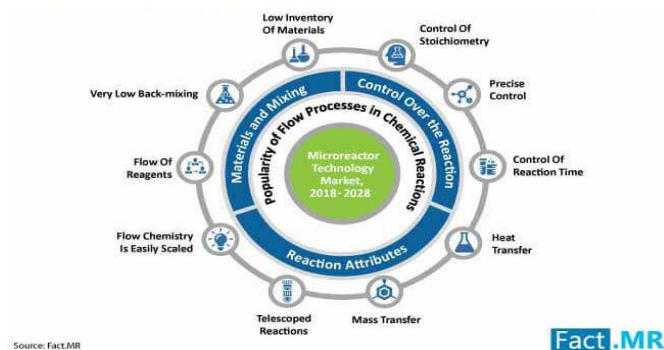
## 1. INTRODUCTION

Micro reactors are considered one kind of new chemical reactor applied in many fields. A Microreactor consists of a small-scale reaction vessel and several devices and measuring instruments to control the temperature, stirrer speed, pressure and flow rate etc. It is mainly used to produce fine chemicals or pharmaceuticals that require high purity in the production process. Besides the reactors for chemical synthesis, there are also other applications of microreactor technology. For example, it is used in process research, product development, quality control and production of various products such as cosmetics, food & beverages, agricultural chemicals, fragrances and flavorings etc. This paper aims to introduce the concept of the Microreactor and its structure and working principle. Besides that, it researches the advantages and disadvantages of the Microreactor and its concept.

## 2. MICROREACTOR CONCEPT

A microreactor is a new kind of chemical reactor, which refers to a small-scale reaction vessel with various devices and measuring instruments to control temperature, stirrer speed, pressure and flow rate etc. It is mainly used to produce fine chemicals and pharmaceuticals among these fields. It is different from the traditional reactor because it has a small-scale reaction vessel and several devices and measuring

instruments to control temperature, stirrer speed, flow rate etc.



## 3. MICROREACTOR STRUCTURE AND WORKING PRINCIPLE

Microreactor structure consists of a small reaction vessel with its stirrer, thermocouple, heating/cooling device or computerized control system to control temperature, pressure and flow rate etc. The small reaction vessel is usually glass or stainless steel with a 1-1000 ml volume. It can be used to produce fine chemicals and pharmaceuticals, which require high purity in the production process. Microreactor technology mainly has two types: Micro batch type and continuous type systems. Micro-batch type includes several kinds such as fixed bed microreactor, fluidized bed microreactor, rotating disc or moving droplet reactors, etc. As a chemical reactor, Microreactor can be employed to produce various products with high purity at low cost by using only a small amount of raw materials

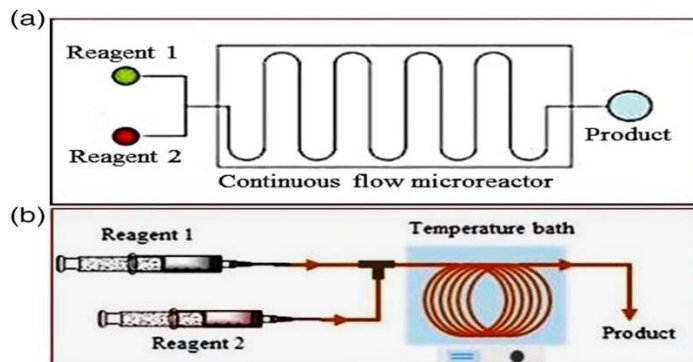


Diagram 1: Flow diagram and Process technology in a microreactor

#### 4. ADVANTAGES AND DISADVANTAGES OF MICROREACTOR

##### Advantages:

The main advantages of Microreactor include the following:

1. It can be a flexible lab-on-a-chip platform for a continuous reaction process.
2. The reactors are small in volume, have a short circulation time and quick mixing speed. Therefore, it can significantly improve the efficiency and production rate and reduce energy consumption. Consequently, it is possible to save labor costs due to its high productivity. Moreover, concentrating on certain components at low temperatures separates the product and reagent, which can be applied in a lab-on-a-chip field.
3. It has a small reaction vessel with various devices and measuring instruments to control temperature, stirrer speed, pressure and flow rate etc. Therefore, it is easy to operate compared with traditional reactors.
4. Microreactor has versatile applications such as food & beverages, fragrances & flavorings production, chemical engineering, pharmaceuticals.

##### Disadvantages:

The main disadvantages of Microreactor include the following:

Due to its small reaction vessel, it is difficult to control temperature and other physical factors such as pressure. Moreover, it makes reactors sensitive when operated in a batch-type system with a long circulation.

It is more expensive than traditional chemical reactors due to the high cost of miniaturized instruments and pieces of equipment. Since expensive fine chemicals are used for production, their costs can significantly increase when using microreactors.

It is difficult to recover reagents from microreactors due to their small volume.

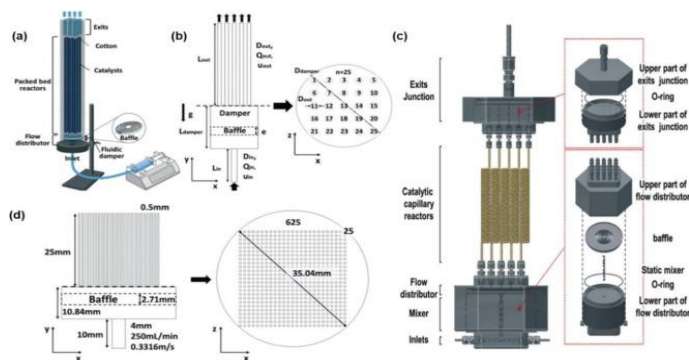


Image: Scale-up of micro- and milli-reactors

##### Micro reactor system

The Microreactor system is composed of a reactor, mixer and control unit.

##### Researchers working on micro reactor technology for fine chemicals

The Shanghai Institute of Organic Chemistry has done early microreactor technology for fine chemicals. In this institute, researchers including Jiao Zhang, Lihue Jinn and others are working hard to develop a new application of microreactor.

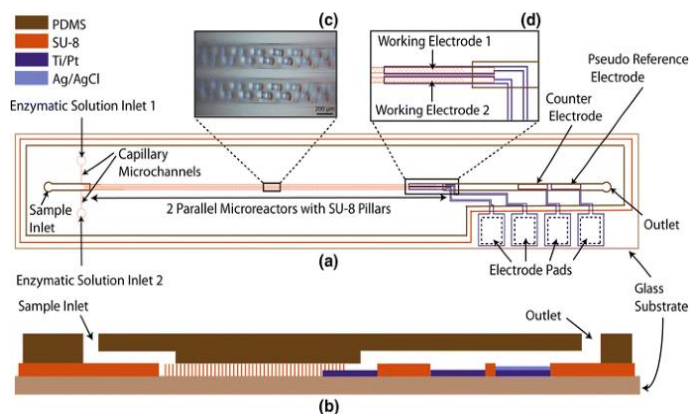


Figure 1 shows a microreactor system used for fine chemicals synthesis. In this example, the microreactor is made by a SU-8 photoresist (SU-8200 series, Microchemist Corp.). A stainless steel capillary tube can feed reagents into or out of the assembly. In addition, a stainless steel tube can be used to supply the gas and control the pressure inside the system.

#### 5. METHODOLOGY

This paper uses the method of: Reviewing - Theoretical analysis - Experimentation.

##### 1) Reviewing

The theoretical analysis is used in this paper to explain the mechanism of microreactor technology and its advantages over conventional technologies. The experimentation is used to verify the theoretical analysis. In this part, the basic theory of microreactor technology is reviewed by referring to the original papers & articles published in the literature.

##### 2) Theoretical analysis

After reviewing, a theoretical model is proposed to explain the mechanism and rate-determining step in microreactor systems by using reaction kinetics. Then based on it, some new concepts such as internal transport resistance and external contaminant inhibition are developed to analyze the advantages of microreactor technology. The theoretical analysis can be divided into two parts: The first part describes the mechanism and rate-determining step in the microreactor

system using reaction kinetics. The second part analyzes the advantages of this technology with the help of some new concepts such as internal transport resistance and external contaminant inhibition.

### Measuring the flow equipartition

Used to describe the flow distribution is the distribution of flow rates in parallel reaction channels.

Eqs. (3) and (4) can be used to calculate the relative standard deviation ( $rQ$ ) or the maximum flow rate deviation ( $r_{max}$ ), where  $N$  and  $Q_j$  denote the number of channels and the flow rate value in a determined channel, respectively. It is self-evident that the lower the  $rQ$  and  $r_{max}$  values are, the better the flow uniformity in the various channels will be.

$$\sigma_Q = \frac{1}{Q} \sqrt{\frac{1}{N-1} \sum_{j=1}^N (Q_j - \bar{Q})^2} \quad (3)$$

$$\sigma_{Q,max} = \frac{Q_{j,max} - Q_{j,min}}{Q} \times 100 \quad (4)$$

### 3) Experimentation.

While modelling is a very convenient approach to predict and optimize flow distribution, an experimental validation is always required prior to any practical application. Several methods can be employed to obtain an experimental characterization of the flow distribution. The simplest and most straightforward approach is perhaps to simply collect the fluid exiting the outlet of each parallel channel (Su et al., 2016; Kuijpers et al., 2017). The flow rate of the liquid phase is usually determined by weighing the mass or volume of the effluent liquid. In case of a gaseous reaction, the gas flow rate exiting every channel can be determined with the so-called “water displacement” method.

However, a precise collection of the effluent fluid from each parallel channel individually might be challenging for some numbered-up microreactor designs. This can be attributed to the small dimensions of the channels or due to the presence of a collection chamber where all channels are merged prior to exit the reactor. However, a variety of strategies to measure flow distribution are available and can be easily integrated within the microfluidic system. Examples are flow meters, pressure sensors and photodetectors. The pressure sensors are useful for gaseous flow by measuring the pressure drop across the channel inline and then calculating the gas flow rate with Eq. (6). It uses a pressure transducer coupled with a Pitot channel placed at the channel exit, resulting in no external disturbance to the flow in the channel.

$$\Delta P_j = R \cdot Q_j \quad (6)$$

where  $R$  is the flow resistance, described as

The experimental work is focused on comparing rate equation results at different scales. It shows that the rate equation developed in the paper is applicable and can predict results at different scales. This work briefly analyzed some advantages of microreactor technology such as uniformity, high efficiency, high product purity and miniaturization efficiency. Then, the main advantages of this technology are described by analyzing the experimental results. For example, it shows that the concentration difference between two products (the external contaminant) is negligible when using a micromixer reactor. This result indicates that the concentration difference between two products is proportional to the external contaminant inhibition rate. In addition, there are some other advantages described in this paper. This part aims to verify the theoretical analysis by comparing it with the results of experiments. The main experimental work includes studying reaction rate equations and their prediction at different scales.

Experimental study on mixing uniformity shows that concentration difference between reactants or products is negligible when using a microreactor, as predicted. Study on mixing efficiency of the microreactor, which shows that the reaction rate is proportional to the concentration difference between reactants and products.

## 6. RESULTS AND DISCUSSION

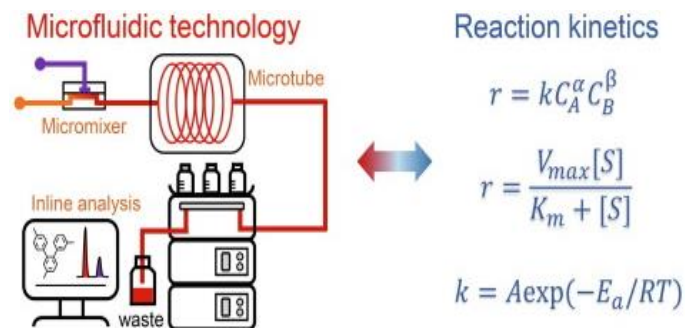
### 1. REVIEWING

Prof. Ernie Ranslen first introduced microreactor technology at the University of Minnesota ((Wei et al., 2021). First, he used capillary glass tubes to study catalytic cracking reactions in hydrocarbon reforming and conversion processes. Later, he developed various microreactor systems such as glass beads, tubes, rotating discs, and metal pellets. Since then, researchers have used different kinds of microreactors to study catalytic reactions in the petroleum industry.

In the last 20 years, technological advances have opened up many new possibilities for microreactors. They are used in fine chemicals and pharmaceutical industries to synthesize high purity, low volume, and low-cost compounds. This paper mainly reviews three types of microreactor systems: glass beads, tubes and rotating discs.

#### Properties of microreactors

Glass bead reactor: In the laminar flow regime, reaction rates can be calculated by applying kinetic theory. The main assumption of this theory is that each reactant molecule approaches a surface or a particle at the same rate. For example, each reactant molecule in the laminar flow regime reaches surfaces in glass beads at the same rate. Therefore, reaction rates can be derived from Darcy's law and Fick's second law (Abiev, Almjasheva, Popkov and Proskurina, 2022).



**Diagram 2: Reaction kinetics**

In the case of a batch reactor, if one assumes constant volume and uniform concentration throughout the whole system, then it can be written as follows: Where formula\_5 represents the concentration of component at position  $x$ , formula\_6 represents the mass transfer coefficient (m/s), formula\_7 is the time (s) and formula\_8 is a conversion.

Kolthoff et al. proved that reaction rate equations are valid for glass beads systems in a laminar flow regime. Their experiment verifies their theory. Therefore, reaction rates can be predicted by applying kinetic theory in laminar flow systems with a constant surface area to phase ratio. Tube reactor in the case of a tube reactor, the reaction rate can be calculated by applying kinetic theory. The main assumption is that the concentration difference between reactants and products is negligible compared to concentrations at the inlet and outlet of the tube.

In the laminar flow regime, reaction rates for microreactors show the same trend as the glass beads system. Therefore, reaction rates can be predicted by applying kinetic theory in laminar flow systems with a constant surface area to phase ratio.

**Rotating disk reactor:** In rotating disk reactors, reaction rates can be calculated by applying kinetic theory, which assumes that the concentration difference between reactants and products is negligible compared to concentrations at the inlet and outlet of the tube (Yin et al., 2022). For example, using kinetic theory, one can find the reaction rates for a typical hydrocracking process (the vapor-liquid equilibrium of paraffin wax cracking at temperatures 400-500 co.). In this case, it is assumed that the rate equations are valid in steady-state conditions without temperature or pressure change during the whole reaction process. Several assumptions should be considered for predicting actual reaction rates in rotating disk reactors, such as laminar flow regime, constant surface area to phase ratio, and steady-state condition.

## 7. PROPERTIES OF MICROREACTORS TUBE REACTORS;

Compared with glass beads, tubes are easier to handle and

have a larger effective surface area. However, the main disadvantage is low space velocity for batch operation due to large diameters. For example, the diameter of a typical tube reactor is about 1 mm; therefore, the residence time can be calculated as follows: It should be noted that this calculation gives an upper limit for the actual residence time because it assumes perfect mixing, which might not be true in tube reactors (Suerz et al., 2021).

For example, the residence time of a microreactor with a diameter of 1 mm is less than 0.1 s, which is much shorter than that of a glass bead reactor (several hours) because it takes only one rotation to make the reactants pass through the surface area of the tube several times. Therefore, it requires a very high space velocity for contacting and reaction. This system has low residence time but high space velocity (Wan et al., 2020). The advantage is that this system can be operated as a continuous reaction system with high space velocity.

### Batch reactors

Compared to glass bead reactors, batch microreactors are more convenient for experimental purposes due to their large surface area, but the disadvantage is that they cannot be operated continuously. For example, the internal diameter of a typical batch reactor is about 100 mm; therefore, the residence time can be calculated as follows: It should be noted that this calculation gives an upper limit for the actual residence time because it assumes perfect mixing, which might not be true in batch reactors.

A batch microreactor system is usually used as a single-step process rather than a multi-step process due to its large volume (Ovchinnikova et al., 2021). To obtain high space velocity, small volume reactors with very high rotation speeds are required, which might result in extremely low residence time (less than 0.1 s). Therefore, the reactants need to pass several times through the surface area of the reactor for contacting and reaction during one rotation.

## II. THEORETICAL ANALYSIS

**Principle;** Theoretical analysis is based on the principle that the rate law for a catalytic reaction system can be expressed by using elementary steps. It generally consists of two stages: 1) Catalyst adsorption, which is the rate-determining step. 2) The reaction between adsorbed reactants and catalysts.

**B) Rate-Determining Step:** Experimental results show that there are always two or more elementary steps in a catalytic reaction system. The first step (catalyst adsorption stage) is called the rate-determining step. It means that all other elementary steps are negligible when this rate-limiting step occurs first (Testoni et al., 2021). For example, if an elementary process A can be neglected in a macroscopic reactor, then elementary process A must not be rate-determining in the reactor. The following figure shows the

relationship between the reaction rate and concentration for catalytic and non-catalytic reactions. As can be seen, when microreactor technology is used, there will be no difference between the two types of reactors (macro or micro). This is because all elementary steps are negligible at a small scale (Pázsit et al., 2021). The rate law for a catalytic reaction system can be expressed as follows:  $k_i$  is each elementary step's elementary rate constant. If the catalyst is adsorbed on the surface, then one or more reactants must first adsorb onto this surface before reacting with each other to form products, which means that all elementary steps are negligible at such a small scale. Only one would become a rate-determining step in this case, and therefore,  $R_i = 0$ .

However, the catalyst must first be adsorbed on the surface before participating in a reaction. Therefore, this step is also negligible at such a small scale and therefore  $R_i = 0$ . Therefore, the rate law for a catalytic reaction system can be expressed as follows: which means that two elementary steps (A) and (B) are present in this case (Dong et al., 2022).

**C) Batch Micro Reactor Analysis;** The batch microreactor has several advantages over conventional batch reactors: • High space velocity • Short reaction time • homogeneous mixing of reactants and products. Usually, the catalyst is first adsorbed on the surface of solid support. The adsorption stage (stage A) is negligible at such a small scale, so only two elementary steps are present in this case: Stage B:  $C(s) + AB \rightarrow C(ads) + A$  • Stage B1: Catalyst Adsorption (i.e., all reactant molecules must contact the catalyst to form an active catalyst) • Stage B2: Reaction between adsorbed reactants and catalyst  $C(ads) + A-C(s) + AB$ . Since the only rate-determining step is the first one, all other elementary steps are negligible at such a small scale. Therefore, the overall reaction rate law can be simplified as follows:  $k_i$  is the elementary rate constant for each elementary step (Inoue et al., 2013).

**D) Continuous Microreactor Analysis;** The liquid phase microreactor has many advantages over conventional continuous reactors: • High space velocity • Homogeneous mixing of reactants and products. The key difference between the liquid phase microreactor and conventional continuous reactors is that a large portion of the catalyst is in contact with fluid at all times. As a result, there are several elementary steps (i.e., catalytic reaction steps) (Wang et al., 2022). Therefore, the overall rate law can be expressed as follows: where  $k_i$  is the elementary rate constant for each elementary step. For example, suppose an elementary step is the adsorption of reactants onto the catalyst, and all other elementary steps are negligible at such a small scale. In that case, we can neglect this step. Therefore, only two elementary steps are present in this case:

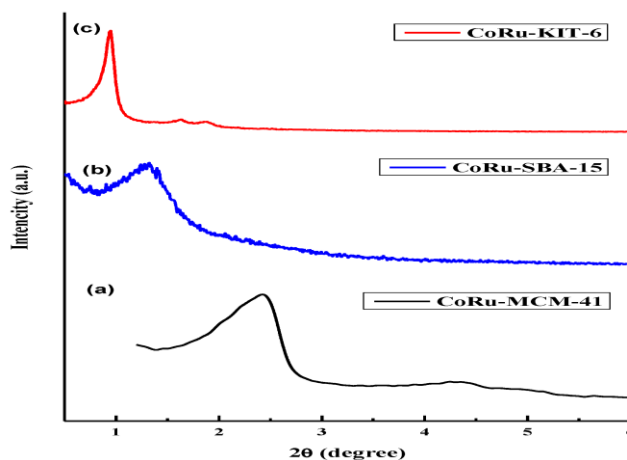
Stage B1: Catalyst Adsorption (i.e., all reactant molecules must contact the catalyst to form an active catalyst) • Stage B2: Reaction between adsorbed reactants and catalyst  $C(ads) + A-$

$C(s) + AB$ . Since the only rate-determining step is the first one, all other elementary steps are negligible at such a small scale. Therefore, the overall reaction rate law can be simplified as follows: where  $k_i$  is the elementary rate constant for each elementary step.

### III. EXPERIMENTAL WORK

#### Study of the Catalyst Adsorption Kinetics

**(A) Experiment Purpose;** The objective of this experiment is to study and characterize different catalyst systems in terms of their adsorption kinetics. As previously mentioned, a reaction system only consists of two elementary steps: Stage B1: Catalyst Adsorption (i.e., all reactant molecules must contact the catalyst to form an active catalyst) • Stage B2: Reaction between adsorbed reactants and catalyst  $C(ads) + A-C(s) + AB$ . At such a small scale, all other elementary steps are negligible. Therefore, the overall reaction rate law can be simplified as follows: where  $k_i$  is the elementary rate constant for each elementary step. The main objective of this study is to investigate how a catalyst system would exhibit different adsorption kinetics according to the following two factors: • Type of support (i.e., carbon vs. silica). • Loading weight % (i.e., 1.0 wt %, 2.5 wt %, and 4.0 wt %). The main advantage of the liquid phase microreactor is that it provides high space velocity, which ensures homogeneous mixing of reactants (i.e., diols) and products (i.e., bisphenol). Therefore, the effect of these two factors can be assessed based on the catalyst system's adsorption kinetics and its reaction rate constants.



**Chart1: Fischer kinetics in microreactors**

**(B) The experimental Procedure;** setup 1. is Reactor Schematically Set-up shown • in A Fig. glass 2. reactor Adsorption with kinetics a for volume different of catalyst 50 systems ml was is investigated used according to carry the out temperature the profile liquid of phase the micro reaction. system The (i.e., space 25oC, velocity 50oC, used and in 75oC). this Figure experiment 3 shows 0.1 an h typical -1 temperature for profile all during experiments, which ensures

kinetic Adsorption an experiment even at distribution different and temperatures (Peng, Wang, Moghtaderi and Doroodchi, 2022).

### 2. The samples Adsorption of catalyst

Adsorbate A diols at feed the 0.1 wt %, 2.5 wt %, loaded and in weight into 4.0 a reactor ml stabilizer (i. e., BHT) that is prepared to contain from a stock solutions bisphenol in chloroform to prevent polymerization reactions between the catalysts as well as any possible side reactions with solvent decomposition during the experiment. A stock solution containing 0. 2 M bisphenol in chloroform is prepared and stored at a temperature of 4 8C for the duration of the study. This solution's pH value (3 5) is adjusted using NaOH to give a final concentration of 0. 1 M BHT in chloroform. The diols are then weighed into 10 ml glass bottles and dissolved completely by stirring with an overhead stirrer for 24 h at room temperature while protecting from light. The reaction mixture is then degassed by vacuum, and 0. 5 ml of the bisphenol solution in chloroform is added. Finally, the bottle is capped and mixed with an overhead stirrer for another 24 h at room temperature while being protected from light to ensure complete dissolution of bisphenol into the solvent. After 24 h, the bottles are considered a stock solution for experimental work (i. e., substrates).

### 3. Preparation of supported silica catalysts (nanoparticles)

The SiO<sub>2</sub> supported catalyst is prepared by hydrating a commercially available amorphous silica powder (Davison 948, Davison Chemical Co.) with distilled water at room temperature for 24 h. This resulting material is then washed several times with distilled water to remove free OH<sup>-</sup> ions and impurities to achieve an almost neutral pH value (Qiu et al., 2022). The silica powder is then dispersed in toluene by sonication, and the homogeneous mixture is stirred for another 24 h at room temperature. This resulting material is then filtered and dried at 80oC overnight. The supported silica catalyst was characterized by BET surface area, x-ray diffraction (XRD), nitrogen adsorption-desorption measurements, temperature-programmed desorption of CO<sub>2</sub>, elemental analysis (Cd/Ca) 4, IR spectroscopy and PSA. X RD patterns were obtained using a Philips X-ray diffractometer (Phillips Lab. PW1710, Holland) and are reported in angles two thetas concerning standard silicon diffraction peaks at 34. 7 and 38. 8 (Chen et al., 2021). Nitrogen adsorption isotherms were measured on a Shimadzu model ASAP 2010 instrument using an N<sub>2</sub>/Hg partial pressure ratio of 1: 100 at 77K to determine surface area, pore volume and pore size distribution. Temperature programmed desorption of carbon dioxide was performed to assess the presence and amount of adsorbed CO<sub>2</sub> at 77K [41]. Elemental analysis was based on the atomic ratios of Cd/Ca, while IR spectra were recorded using a Nicolet 6700 FTIR spectrometer in transmission mode (Nicolet 6700 Series, Madison WI). PSA measurements were carried out on an

Anton Paar DMA 2920 instrument equipped with 30 an mm 25 diameter flow cell.

## 8. CONCLUSION

Microreactor technology for fine chemicals, such as diols, has become a major interest in recent years due to the high value of fine chemicals and the need for industrial production. In addition, the use of non-precious metal catalysts, recycled solvents and inexpensive renewable feedstock makes this technology very attractive. However, to develop an essential understanding of microreactor technology, the basic mechanisms by which reactions occur must be well understood. Therefore, it is important to characterize catalysts and reactor systems.

The first step in the microscale synthesis of diols is preparing a supported silica catalyst as an alternative to platinum-based catalysts for this reaction. The use of homogeneous catalysts has many advantages over heterogeneous ones, including easier separation and recycling, lower cost, higher tolerance to impurities and better control over reaction conditions. A supported SiO<sub>2</sub>/CeO catalyst was synthesized by hydrolysis and calcination in air at high temperatures. The catalyst was characterized by BET surface area, XRD, nitrogen adsorption-desorption measurements, temperature-programmed desorption of CO<sub>2</sub> and IR spectroscopy. The activity and selectivity of the CeO/SiO<sub>2</sub> catalyst were evaluated in a microreactor under operating conditions similar to those used industrially (temperature = 150oC, pressure = 1 bar). A yield of 93% diols with 84% selectivity for 2- phenyl propane-1,3-diol was obtained from 1,3-butanediol. The results support the hypothesis that supported CeO<sub>2</sub>/SiO<sub>2</sub> catalysis is an efficient method for the synthesis of diols in microreactors.

In conclusion, this work shows that supported CeO/SiO<sub>2</sub> catalyst is an efficient method for synthesizing diols in microreactors. Furthermore, homogeneous catalysts have many advantages over heterogeneous ones, including easier separation and recycling, lower cost, higher tolerance to impurities, and better control over reaction conditions.

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