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Research Paper

Efficient Ibuprofen synthesis from isobutyl-benzene under ambient conditions

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ABSTRACT

Ibuprofen is classified as a non-steroidal anti-inflammatory medication (NSAID). This medication is employed for the treatment of several ailments, encompassing mild to moderate pain such as toothache, migraine, and period pain. Additionally, it is used to alleviate high-temperature fevers resembling influenza, reduce inflammation in tissues, and manage conditions like rheumatoid arthritis, back pain, and gout, among others. Despite the relative simplicity of ibuprofen's structure, it possesses significant structural complexity to enable a broader variety of acquisition methods. Numerous techniques for acquiring it with possible industrial utility have been discovered since the inception of its initial commercial use. The majority of commercially viable industrial processes utilized for the synthesis of ibuprofen are derived from isobutyl-benzene (IBB). Commercial isobutyl benzene (IBB) is synthesized through the reaction between propene and toluene. The conversion of isobutyl benzene ranges from (18.05%) to (19.8%) at various values of reaction temperature and from the results we can see that the reaction time has a signifi-cant effect on the isobutyl benzene conversion.

1. Introduction

The substance is isobutyl benzene, which possesses the molecular formula C10H14. The material under examination is a liquid that is organic, odoriferous, colorless, and neutral. It is commonly referred to as 2-methyl-1-phenylpropene. This substance holds significant importance as an organic component and finds extensive application in the pharmaceutical and fragrance sectors [1]. IBB is a prominent primary ingredient used in the pharmaceutical and fragrance sectors because of its delightful, sweet scent commonly associated with cherry, with a faint undertone of balsamic qualities, [2]. The molecule is synthesized through the process of side-chain alkylation, wherein toluene and propylene undergo a reaction in the presence of an alkali metal catalyst. More specifically, this particular reaction employs potassium metal as the catalyst, [2]. The global perfume business extensively utilizes isobutyl benzene as a superior-grade raw ingredient. Furthermore, the industrial production of this renowned medicinal compound, well regarded for its anti-inflammatory and analgesic properties, widely employs ibuprofen, [3]. Ibuprofen is a commonly preferred choice for treating several diseases, including pain relief from headaches, muscle pains, and menstrual cramps, as well as regulating body temperature. Prophylaxis for inflammatory conditions like arthritis and gout [4]. It is expected that the global ibuprofen market will reach a value of over $630 USD Million in 2022, exhibiting a compound yearly growth rate (CAGR) of 2-5% shortly. Notably, this market identifies North America, Europe, and China as major markets, [5]. Medicine's affordability and accessibility are increasing, [6]. The synthetic synthesis of ibuprofen involves the utilization of the acylation process, which involves the reaction of isobutyl benzene. In the production of ibuprofen, the initial step involves an acylation procedure employing the Friedel-Crafts reaction, [7]. Because of this reaction, isobutyl benzene (IBB) is mixed with acetic anhydride, which acetylates it. This creates 4-isobutyl acetophenone (4-IBAP). Hydrofluoric acid facilitates the Friedel-Crafts acetylation reaction, acting as a highly effective Lewis's acid essential for the reaction. In the first step, 4-IBAP is mixed with hydrogen in the presence of an H_2 catalyst. This makes 1-(4-isobutyl phenyl) ethanol. The synthesis of ibuprofen, the final product, is achieved by the utilization of a carbonylation method involving 1-(4-isobutyl phenyl) ethanol [8, 9]. To mitigate the expenses and time requirements associated with creating new pharmaceutical substances, a feasible option is to improve the effectiveness of current medicines by administering them in a controlled manner with precise targeting of their administration [10, 11]. Diffusion and chemical reaction mechanisms within the boundary layer interact concurrently to cause mass transfer, which in turn determines the overall rate [12, 13]. There are several well-established and cutting-edge techniques for synthesizing ibuprofen. The Boot method and the Hoechst process are widely recognized and established techniques for the acquisition of ibuprofen [14]. The Boots Method, sometimes known as the Browns Method, is a patented technique used for synthesizing ibuprofen. It is widely recognized as the standard approach in the field. The research employs a methodology consisting of six phases in the synthesis process, commencing with the utilization of the chemical 2-methylpropylbenzene [15]. It should be emphasized that this chemical compound might be derived from different constituents taken from crude oil [16]. The molecule exhibits a carbon structure that bears resemblance to that of ibuprofen. The Boots method, which has been widely employed in the industrial production of ibuprofen for a significant period, has effectively yielded substantial volumes of ibuprofen, surpassing hundreds of metric tons over the course of the last forty years [17]. However, this methodology has also generated a significant number of chemical by-products that are unsolicited, unnecessary, and not eligible for recycling [8, 18].

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Nomenclature:		BHC	Browns Hoechst Company
List of variables:		HF	Hydrofluoric acid
A	Agitation rate (rpm)	IBB	Isobutylbenzene
T	Temperature (C°)	NSAID	Non-steroidal anti-inflammatory drugs
t	Time (hr)	2-(4-IBP)	2-(4-Isobutyphynyl)
List of parameters:		4-IBAP	4-Isobutylacetophenon
BBD	Box-Behnken Design		

Since then, scientists have modified the Hoechst process and officially referred to it as the green synthesis process [19]. The team of researchers in Bangladesh has selected the BHC reaction route as the preferred method of production. The key components of this pathway include IBB, $C2H6O, C4H6O3, HF$ acid, H_2, and CO. The hydrofluoric acid, acetic anhydride, and isobutyl benzene are delivered into PFR, To maintain a temperature of approximately 80C°and a pressure of around 700kPa, the reactor is outfitted with a water-cooling system. 4-isobutyl acetophenone is made when isobutyl benzene reacts with acetic anhydride in the presence of hydrofluoric acid. The Plug Flow Reactor (PFR) produces a continuous flow of output [20]. Under the influence of pressure and heat, a plug-flow reactor (PFR) is observed.

1.1 Aim of study

Production of ibuprofen through the Friedel-craft acylation of isobutyl benzene under ambient conditions, the experiments were conducted by varying (reaction temperature, agitation rate, and reaction time) temperature of 25C°, the exit stream is directed into a 3-phase separator. In this separator, the stream is separated into two phases: one containing a high concentration of water and the other containing a high concentration of 4-isobutyl acetophenone. The phase containing 4-isobutyl acetophenone is subsequently placed into a series of distillation columns at a temperature of 25C°to remove any impurities [21], and 655 kilopascals.

The resulting product, which has a concentration of more than 99% of 4-isobutyl acetophenone by weight, is then cooled to a temperature of 90C°. The stream of nearly pure 4-isobutyl acetophenone is then supplemented with hydrogen gas. Afterward, the mixture is cooled and then transferred to the fixed-bed reactor, where the chemical reaction between 4-isobutyl acetophenone and hydrogen occurs, leading to the production of 1-(4-isobutyl phenyl) ethanol. The experimental procedure entails the amalgamation of carbon monoxide with a stream of 1-(4-isobutylphenyl) ethanol, followed by the application of heat to a temperature of 120C°. Subsequently, the resultant stream is conveyed to a plug-flow reactor encompassed by cooling coils. The reaction between 1-(4-isobutyl phenyl) ethanol and carbon monoxide in the reactor results in the formation of ibuprofen, the ultimate product. The [22, 23], each non-steroidal anti-inflammatory medicine (NSAID) can elevate blood pressure levels. It is important to note that the average increase in blood pressure is modest, although its impact exhibits significant variability across individuals.

The Joint National Committee's report reveals a correlation between the use of non-steroidal anti-inflammatory drugs, such as ibuprofen and similar medications, and the development of high blood pressure and resistant hypertension, When administered in the appropriate dosages to alleviate pain or mitigate inflammation, ibuprofen has an impact on blood pressure. Ibuprofen influences blood pressure through various methods, one of which is its ability to mitigate the impact of drugs used to treat hypertension. Water retention increases while sodium excretion decreases. The phenomenon leads to an elevation in bodily fluid retention and has an impact on renal function, resulting in hypertension and heightened strain on the cardiac and renal systems.

The objective is to enhance the likelihood of experiencing a heart attack or stroke, particularly while consuming elevated quantities of ibuprofen or other non-steroidal anti-inflammatory medicines, To mitigate the impact of ibuprofen on blood pressure, it is advisable to engage in a consultation with a specialist physician regarding suitable analgesic alternatives, encompassing the subsequent options. Before taking paracetamol or aspirin, it's best to consult a doctor, especially if you have gastrointestinal illnesses, gout, liver disorders, or other similar conditions [24]. Studies have continued to develop methods for producing ibuprofen since its discovery by the scientist David, who considered the discovery of this drug a qualitative leap in the field of anti-inflammatory drugs. The main goal was to discover better methods in terms of low cost, quick production time, and most importantly environmentally safe, however after the emergence of the Corona epidemic, the killer: The attention of scientists and discoverers turned to find a medicine or vaccine to confront this epidemic. Unfortunately, this increased the monopoly of the companies that manufacture ibuprofen and other important medicines. Thus, this work is re-examined the most important method of producing ibuprofen and found solutions to the most important obstacles in a way that suits the user.

2. Experimental work

The materials used can be in this work found in Table 1. The apparatus used here consisted of a 250 ml glass batch reactor with three necks, a Reflex condenser, a Hot plate magnetic stirrer, a thermometer, and a Laboratory stand.

Table 1. The list of materials.

Materials	Properties	Manufacturing
Isobutyl-benzene	1. Purity (98%) 2. Density (0.853g/ml) 3. Mw (134.22)	Manufactured by Shanghai Macklin Biochemical Company Ltd-China.
Acetic Anhydride	1. Purity (99.8%) 2. Density (1.082g/ml) 3. Mw (102.089)	Manufactured by Flucka Company.
Hydrofluoric Acid	1. Purity (99.9%) 2. Concentration (48%) 3. Mw (20.01)	Manufactured by Sisco Research Laboratory Pvt Ltd(SRL)-India.

Mw:Molecular weight

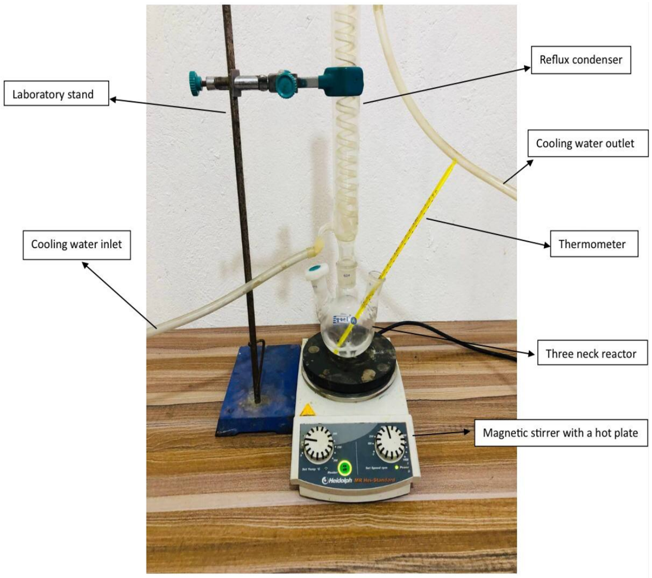

Figure 1. The experimental apparatus for this study.

2.1 Experimental design

RSM (Response surface methodology) was the statistical methodology used to optimize the production of ibuprofen from isobutyl benzene by Minitab 18 software. An approach to enhance efficiency in the process of research and development, aiming to generate high-quality goods promptly and at a reduced expense [25]. The use of this methodology requires the selection of appropriate responses (isobutyl-benzene conversion), variables, and levels. Variables were temperature, agitation rate, and the reaction time listed with their levels in Table 2 by (BBD) box-Behnken design, according to the BBD matrix, fifteen experimental run variables were conducted.

Figure 1 shows the experimental setup for this process. The Production process in this study is carried out according to the following steps listed in [22], which are also shown in Fig. 1. First, the reaction system is prepared for work. The (4 *ml*) isobutylbenzene is measured and added to the glass flask. After the (4 *ml*) acetic anhydride is added to isobutylbenzene, the (5 *ml*) hydrofluoric acid is added as a catalyst. After completing the addition of all materials, the reaction system is prepared for work. Set the reaction temperature, reaction time, and agitation rate according to the data of each experiment (based on the design of the experiments in the Minitab 18 software). Given that the boiling point of hydrofluoric acid is 19.5° C. The condenser is operated using cold water throughout the experiment to prevent the hydrofluoric acid from evaporating.

Table 2. Process variables with their levels.

Variables	Minimum	Middle	Maximum
Temperature (C°)	25.00	27.50	30.00
Time (hr)	01.00	02.00	03.00
Agitation rate (rpm)	100.00	550.00	1000.00

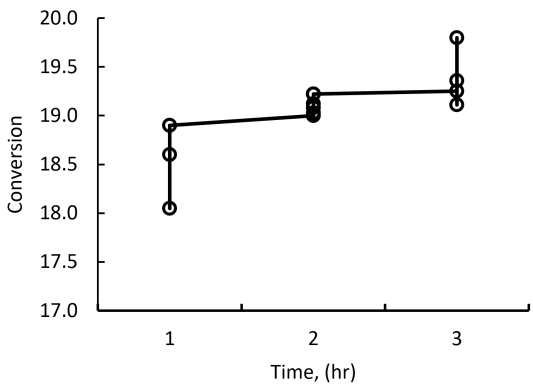

Figure 2. The effect of time on the isobutyl benzene conversion.

3. Result and discussion

The conversion of isobutyl benzene was in the range of (18.05% - 19.8%). As a preliminary inspection, a comparison between run (1) and run (7) showed that the agitation rate has a considerable impact on the conversion where from (18.60%) to (19.08%) making a difference of (0.48%) as an agitation rate increased from (550 *rpm*) to (1000 *rpm*).

3.1 The Influence of Process Factors on the Conversion of isobutylbenzene

A catalyst speeds up the chemical reaction between isobutyl benzene and acetic anhydride, which is what makes ibuprofen. The final result of this reaction is the formation of ibuprofen and acetic acid as final products. The reaction rate of this particular chemical reaction is significantly influenced by the presence of reactants at the active sites of the catalyst. It is feasible to manipulate multiple variables, including the reaction temperature, reaction time, and agitation rate.

3.1.1 Effect of time on the isobutyl benzene conversion

An essential factor that will impact the production of ibuprofen is reaction time, the optimal value of reaction time is 3 *hr*, as shown in Fig. 2, the relation between the isobutyl benzene conversion to produce ibuprofen and reaction time. It is noted that the reaction time has a clear effect on the conversion rate and accelerates the reaction rate, as the reaction time rises, the conversion increases.

3.1.2 Effect of temperature on the isobutyl benzene conversion

The isobutyl-benzene conversion ranged from (18.05% to 19.8%). The reaction takes place within a temperature range of 25–30C°. The temperature exerts a subtle yet tangible influence on the chemical conversion of isobutyl-benzene. In addition, a reflux condenser was included in the experiments to minimize the loss of hydrofluoric acid due to its boiling point being exceeded by the reaction temperature 19.5C°. Figure 3 illustrates the effect of temperature on the conversion, this figure shows that temperature has a slight but somewhat significant effect on the conversion of isobutyl benzene.

3.1.3 Effect of agitation rate on the isobutyl benzene conversion

An agitation rate was noticed to have a constant effect on the conversion of isobutyl benzene, the percentage of the conversion obtained at (1000 *rpm*) reached (19.8%). Figure 4 shows a considerable rise in isobutyl benzene conversion, increasing agitation rate has a positive effect on the isobutyl benzene conversion at a high temperature and time.

3.2 Optimization parameters

Figure 5 shows the operational variable's ideal values; The conversion increases to 19.8% with an increase in time, agitation rate, and reaction time, hence the optimal time was the highest value that could be achieved. The optimal conditions obtained can be used to improve the efficiency and cost-effectiveness of the process.

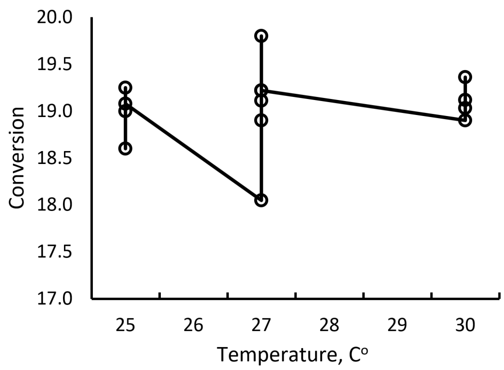

Figure 3. The effect of temperature on the conversion.

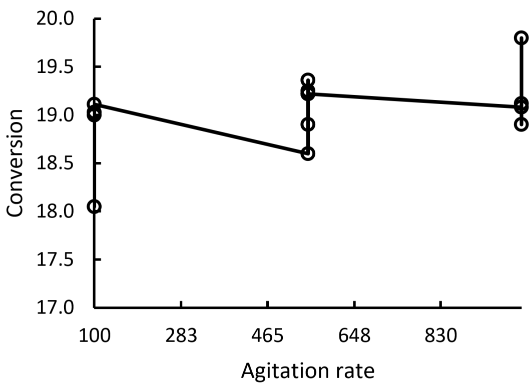

Figure 4. The effect of agitation rate on the conversion.

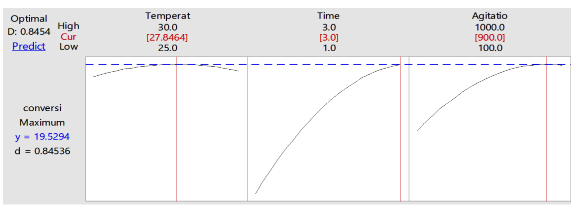

Figure 5. The optimal values of the operating variables.

4. Conclusions

Ibuprofen, a nonsteroidal anti-inflammatory medicine (NSAID), is well acknowledged for its therapeutic efficacy in relieving pain, inflammation reduction, and the treatment of many different medical problems. Ibuprofen can be synthesized by reacting isobutyl benzene with stearic anhydride in the presence of hydrofluoric acid as a catalyst at atmospheric pressure and temperature to get isobutyl-benzene conversion about (18.05% - 19.80%). The impact of time on the conversion of isobutyl-benzene is widely recognized, with temperature and agitation rate having a minor influence but also notable.

Authors' contribution

All authors contributed equally to the preparation of this article.

Declaration of competing interest

The authors declare no conflicts of interest.

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Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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