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Sonochemical Formation of Peracetic Acid in Batch Reactor: Process Intensification and Kinetic Study

Prashant D. Jolhe, Bharat A. Bhanvase, Satish P. Mardikar, Vilas S. Patil and Shirish H. Sonawane

Abstract

The present chapter highlights the kinetic studies for the sonochemical synthesis of peracetic acid (PAA) in a batch reactor. The effect of different operating parameters including acetic acid to hydrogen peroxide molar ratio, temperature, catalyst loading, effect of ultrasound, were studied using Amberlite IR-120H as a catalyst. The deactivation of the Amberlite IR-120H catalyst has also been studied. The experimental data were further utilized for the estimation of intrinsic reaction rate constants and equilibrium constants. From the experimental results; the optimized PAA concentration was observed for 471 mg/cm³ catalyst loading at 40°C with acetic acid to hydrogen peroxide molar ratio equals to 1:1 in the presence of ultrasound. Results also revealed that the reaction rate was found to be significantly enhanced in the presence of ultrasound, which can be attributed to the enhanced mixing and in-situ formation of H₂O₂. The use of ultrasound drastically reduces the overall reaction time to 60 min, which is very less compared to 30 h as reported for conventional batch reactor utilizing H₂O₂ only.

Keywords: peracetic acid, ultrasound, kinetics, batch reactor, kinetics

1. Introduction

Peroxy-carboxylic acids are widely employed as disinfecting/antimicrobial agents and thus hold great industrial importance [1]. Besides these properties, these are also utilized as bleaching agent for wood pulp in paper industries, for Baeyer-Villiger synthesis of fine chemicals and olefins epoxidation. Exceptional oxidative properties of peroxy-carboxylic acids offer green alternative to conventional methods employing chlorine dioxide owing to nontoxicity of their decomposition products [2].

Although peracetic acid and peroxy-propionic acids are widely employed peroxy-carboxylic acids, less stability and their explosive nature often possess difficulties in their synthesis. Generally, PAA can be prepared by two ways either from H₂O₂ or by oxidation of acetaldehyde which can be carried out in liquid or vapor phase [3]. The former way of liquid-phase synthesis is being used for several years due to comparative safer operations [1]. However, it suffers from slower reaction

rates and thus requires involvement of catalysts. Industrial production of PAA is still being carried in the presence of sulfuric acid as acid catalyst [4, 5]. The utilization of sulfuric acid brings lot of challenges in view point of corrosion, environmental threats, and post-reaction catalyst recovery. Sustainability of industrial production is generally based on safety aspects of the production of chemicals and their environmental impact. The concept of green chemistry offers solution for prevention of waste products, suppresses the energy consumption, and provides safer processes with optimized productivity. Process intensification as one of the tool of green chemistry is any improvement made in chemical process that gives a considerable cleaner and more energy solving technology [6].

Several approaches have been adopted by many researchers for the process intensification in chemical process in industries. Recently, ultrasound has proven to be a promising tool for process intensification leading to enhanced reaction rates and mass transfer. The use of ultrasound offers various applications, which includes cleaning, organic synthesis, catalysis, extraction, emulsification, material processing, food processing, waste-water treatments, etc. [1, 7]. Ultrasonic waves cannot couple directly with molecular energy levels. The influence of ultrasound on a chemical reaction is attributed to the formation of cavitations. So, as it is based on the important phenomenon of cavitations which effects generation of extreme conditions of very high temperatures and pressures (1000 atm pressure and 10,000 K temperature) locally with overall ambient operating conditions. The formation of cavitations depends not only on ultrasonic power applied but also on physical properties of irradiated liquid rather than on chemical properties. Under ultrasound treatment, the course of chemical reaction can be influenced by two ways: mechanically due to streaming caused by a collapse of cavitation and by the formation of reactive species of volatile substances in the medium [8]. Due to this phenomenon, release of highly reactive free radicals, generation of turbulence and liquid circulation (acoustic streaming) takes place which in turn enhances the rates of transport processes [9]. These above mentioned effects of ultrasound have a several extensive applications in the areas of chemical processing that result in the decreased reaction time, increased reaction yield, and increase in the effectiveness of the catalyst [10–13].

Most common method for synthesis of peroxy-carboxylic acids (PCA) is the oxidation of the parent aldehyde or carboxylic acid [14]. Generally, acid catalysts are used either in homogeneous or heterogeneous forms during the synthesis of peroxy-carboxylic acids. Furthermore, several techniques for the preparation of per carboxylic acids have been reported in the literature that uses the batch process [3, 15, 16]. However, the formation of peroxy-carboxylic acid is limited due to its unstable nature and reversible hydrolysis reaction. Additionally, the drawbacks of using sulfuric acid are corrosion of the equipment, setup of an energy-consuming distillation system for the catalyst separation, and threat to the environment [17]. Therefore, more research attention is being driven to the development of heterogeneous catalysts. The use of ultrasound during the preparation of peroxy-carboxylic acids is an important issue of investigation.

Ultrasound irradiation has been proved to be environmentally benign method to process several chemical reactions [18, 19]. The use of ultrasound enhances the reaction rate which is supposed to be due to H_2O_2 generation and intense mixing which enhances the mass transfer rate [20]. Considering the above discussion herein present report, we are demonstrating the synthesis of PAA in presence of ultrasonic irradiations. The cation-exchange acid resin- Amberlite IR 120H was used as catalyst. The effects of several parameters such as molar ratio of acetic acid to hydrogen peroxide, temperature, catalyst loading, and ultrasonic irradiations were investigated.

2. Experimental

2.1 Materials

PAA was synthesized using anhydrous acetic acid and hydrogen peroxide (30 wt. %) (S.D. Fine Chem.) in presence of cation-exchange resin as a catalyst. Cation-exchange resin (Amberlite IR-120H, mean diameter = 0.83 mm, density = 1.28 g/cm [3]) was purchased from Rohm & Haas. Analytical grade ceric sulfate, potassium iodide, and sodium thiosulfate were procured from Merck and were used as received. Sulfuric acid solution, starch indicator solution, and ferroin (1, 10-phenanthroline ferrous sulfate, 0.01 molar in water) solutions were used for the analysis of PAA. Deionized water was used in all the experiments.

2.2 Experimental setup and procedure

The experimental setup used for the synthesis is as shown in **Figure 1**. A round bottom flask of 50 mL was used for all the experiments. The reactor was kept in a commercially available 1.5 L ultrasound bath (Biotechnique Laboratory, Model BTC Sr. No. 10242, frequency = 40 kHz, power = 150 W, amplitude = 15) equipped with external cryostat for maintaining a constant temperature (20–40°C). During the course of reaction, 50 mL round bottom flask containing 13.4 mL of reaction mass was irradiated in ultrasound bath. The cation-exchange capacity of Amberlite IR-120H by dry weight (meq/g) was used to calculate the apparent $[H^+]$.

In a typical reaction, 4.5 ml (0.074 moles) of glacial acetic acid and 8.9 ml (0.078 moles) of 30% H_2O_2 solution were thoroughly mixed in a 50 mL round bottom flask. To this solution, certain amount (0.75–1.5 g) of dry catalyst, that is, Amberlite IR-120H was added. The resultant reaction mixture was then treated for 60 min using ultrasound irradiation (40 kHz, 150 W). A fixed amount of reaction mixture (0.3 mL) was sampled at regular interval of time and analyzed for the determination of PAA concentration. Mole fractions of H_2O_2 and PAA were determined by the titration method previously reported by Greenspan and Mackellar [21]. The effect of different parameters, namely effect of ultrasound, PAA: H_2O_2 mol ratios, effect of temperature, effect of catalyst concentration, and catalyst deactivation were studied. The molar ratio of acetic acid to hydrogen peroxide was varied from 1:0.45 to 1:2.50. All the experiments were performed in the temperature range of 20–40°C with an interval of 10°C and at the atmospheric pressure.

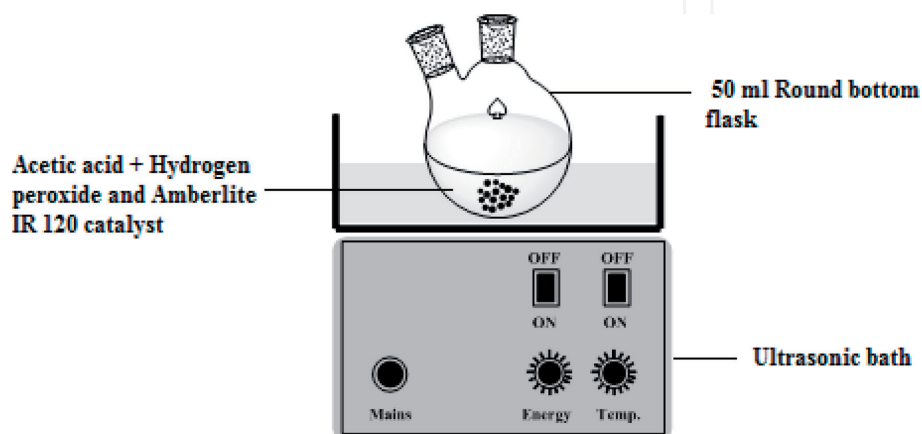


Figure 1.
Schematics of experimental setup for the preparation PAA in batch reactor.

3. Result and discussion

3.1 Effect of molar ratio of acetic acid: H₂O₂ on PAA formation

Figure 2 depicts the effect of different molar ratios of acetic acid to H₂O₂ on PAA formation. From figure, it can be observed that, as the molar ratio of acetic acid to H₂O₂ increases from 1:0.45 to 1:2.50, the PAA formation is found to be significantly increased in presence of ultrasound (40 kHz, 150 W). The energy input is improved by harnessing the power of pressurized water as transfer medium, which transfers the ultrasonic energy indirectly into the microstructured device. The mechanical and vibrational effects of the ultrasound are the main reason of better mixing as it increases the interfacial area between the phases of a heterogeneous system rather than increase in the temperature. These factors might be responsible for the increased concentration of PAA (from 2.125 to 5.2 mol/L) with an increase in the molar ratio from 1:0.45 to 1:2.50 in the presence of ultrasound (40 kHz, 150 W). The increase in the concentration of H₂O₂ increases the probability of substrate-active-site interaction and ultrasound further enhances the rate of reaction. The ultrasound increases the interfacial area between the solid and organic phases and hence the interactions also increase resulting in higher yield of PAA.

3.2 Effect of catalyst loading on PAA formation

Figure 3 depicts the effect of amount of catalyst loading on the formation of PAA in presence of ultrasound irradiation. These reactions were carried at 40°C, while maintaining the molar ratio of acetic acid to H₂O₂ as 1:1. From **Figure 3**, the observed PAA concentration for 340 mg/cm³ catalyst loading was found to be 2.875 mol/L after 60 min. With increase in catalyst loading amount to 707 mg/cm³, PAA concentration was found to increase to 3.375 mol/L. This can be attributed to additional acidic sites accessible at higher loading of catalyst that significantly increases the PAA concentration and equilibrium is reached earlier. The increase in the PAA yield is attributed to the synergic effect of ultrasound. Further, the concentration of PAA at 707 mg/cm³ catalyst loading at 40°C is established to be closer to 3.45 mol/L, which is closer to the PAA concentration obtained at 471 mg/cm³ catalyst loading at the end of 60 min. This indicates that optimum value of the catalyst loading during

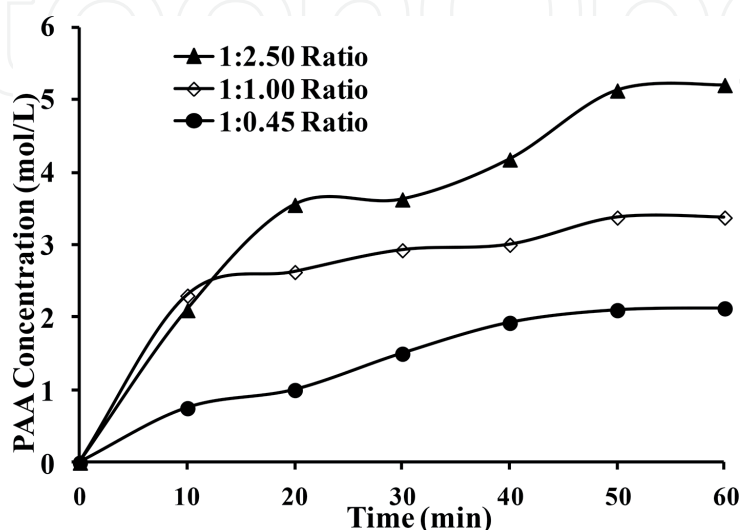


Figure 2. Effect of molar ratio of acetic acid to hydrogen peroxide on PAA formation (catalyst loading = 471 mg/cm³, temperature = 40°C) in batch reactor.

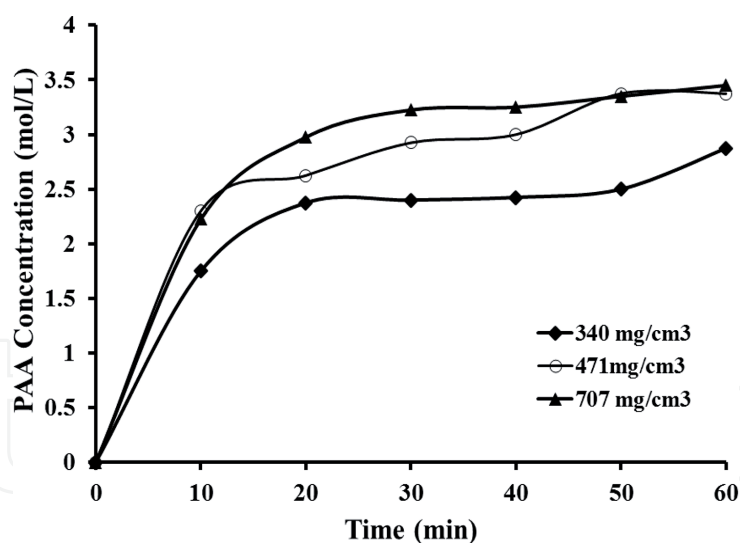


Figure 3.
Effect of Amberlite IR-120H catalyst loading (in mg/cm³) on PAA formation (molar ratio of acetic acid to H₂O₂ = 1:1, temperature = 40°C) in batch reactor.

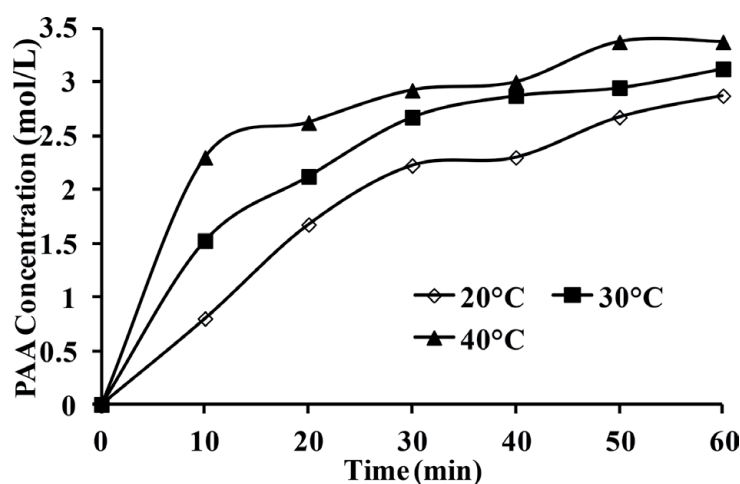


Figure 4.
Effect of reaction temperature on PAA formation (molar ratio of acetic acid to H₂O₂ = 1:1, catalyst loading = 471 mg/cm³).

the formation of PAA in batch reactor in the presence of ultrasound is 471 mg/cm³, which provided adequate numbers of acidic condition. In case of increased catalyst loading, although, large number of acid sites is available at 707 mg/cm³ catalyst loading, the presence of fixed amount of acetic acid limits the formation of PAA in the batch reactor. Therefore, the concentration of PAA remains unaffected. In the current set of the experiments, the equilibrium reaches approximately in 30 min against 30 h in conventional batch reactor indicating faster reaction rate and is obviously attributed to the cavitation effects of the ultrasonic irradiations, which form H₂O₂ due its chemical effect that enhances the reaction significantly.

3.3 Effect of temperature on PAA formation and kinetic study

Figure 4 depicts the effect of the temperature on PAA formation for a molar ratio of acetic acid to H₂O₂ = 1:1 and catalyst loading = 471 mg/cm³ in batch reactor in the presence of ultrasound irradiation (40 kHz, 150 W). It is found that that the concentration of PAA increases to 3.375 mol/L (at 40°C) from 2.875 mol/L (at 20°C) at the end of 60 min. Thus, the reaction rate is enhanced with an increase in the temperature and also with the use of ultrasonic effect. The results are in good

agreement with those of previous results [22]. It has been reported that the rise in the temperature provides the activation energy to the reactant molecule [23], However, too high reaction temperature may lead to decomposition of the product (PAA) leading to low product yield; thus, the experiments were carried while maintaining the temperature at 40°C maximum. Additionally, at higher temperatures, the vapor so formed may get trapped in the cavitating bubbles leading to milder collapse of cavities, which consequently result in lesser formation of H₂O₂ in the reaction medium. At lower temperature (i.e., up to 40°C), the amount of vapor present in cavitating bubble is less due to less vapor pressure. An intense collapse of cavity of the bubble brings about chemical changes, which result in the formation of more amount of H₂O₂ in the reaction medium. As a result of which, net reaction rate is enhanced at moderate reaction temperature (40°C).

Figure 5 depicts the plot of $\ln K_C$ versus $1/T$ for the given experimental data reported in **Figure 4**. The obtained equilibrium constant correlation for the experimental conditions is $\ln K_C = \frac{-2189.4}{T} + 9.3726$ for molar ratio of acetic acid to H₂O₂ = 1:1 and catalyst loading = 471 mg/cm³. Also, **Figure 6** reports the plot of $\ln k_{1obs}$ and $\ln k_{2obs}$ versus $1/T$ for the given experimental data reported in **Figure 4**. The obtained kinetic reaction rate constant correlations are $\ln k_{1obs} = \frac{-4887.8}{T} + 7.2109$ and $\ln k_{2obs} = \frac{-2698.3}{T} - 2.1617$ for molar ratio of acetic acid to H₂O₂ = 1:1 and catalyst loading = 471 mg/cm³. The estimated activation energy for forward reaction is 40.60 kJ/mol and for reverse reaction 22.43 kJ/mol. These activation energy values are significantly higher than previously reported values by Zhao et al. [3], where it takes 30 h to attain equilibrium in batch reactor. In present study, the equilibrium is attained within 30 min, which can be attributed to the cavitation effects of ultrasound which generates excess of H₂O₂ that accelerates the reaction rate.

3.4 Deactivation of catalyst in batch reactor

In present investigation, the deactivation and reusability of cation-exchange resin, that is, Amberlite IR-120H has also been studied. For this, batch reactions were carried in presence of H₂O₂ and ultrasound irradiation (40 kHz, 150 W). The typical experiments were performed at a molar ratio of acetic acid to H₂O₂ = 1:1 with catalyst loading of 471 mg/cm³ and at temperature 40°C. After each

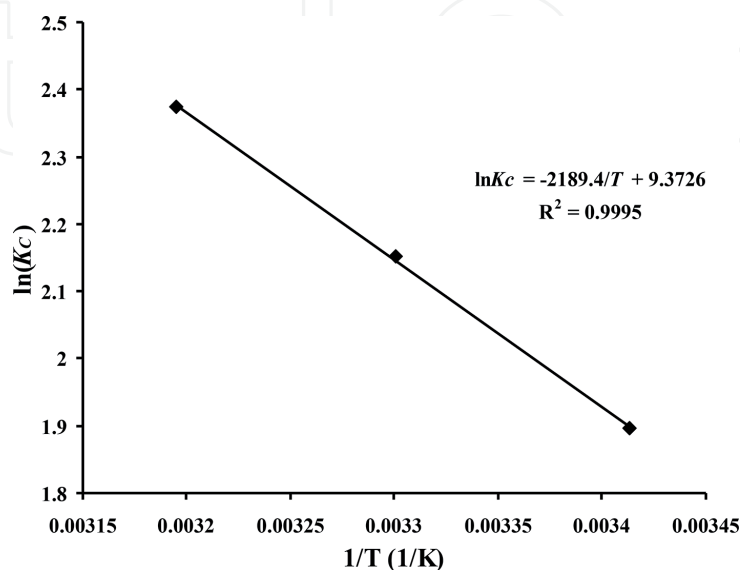


Figure 5. The Arrhenius plot of equilibrium constant at different temperature (in kelvin) for PAA formation (molar ratio of acetic acid to H₂O₂ = 1:1, catalyst loading = 471 mg/cm³).

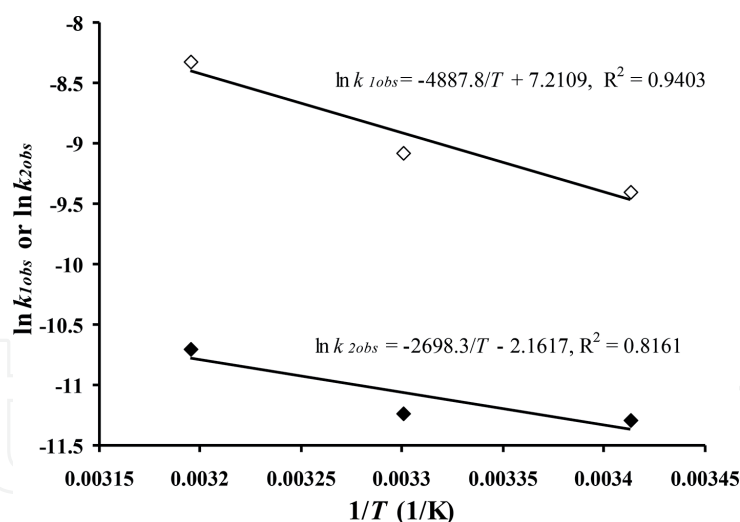


Figure 6. The Arrhenius plots of the intrinsic reaction rate constants at different temperature (in Kelvin) for PAA formation (molar ratio of acetic acid to $H_2O_2 = 1:1$, catalyst loading = 471 mg/cm^3).

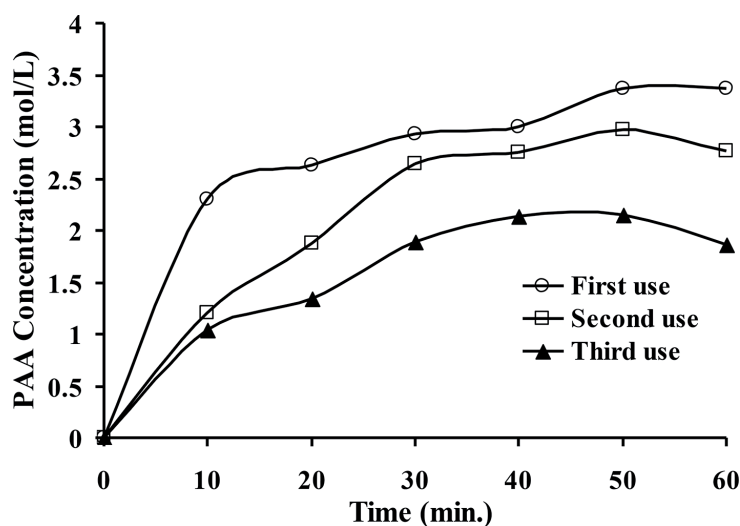


Figure 7. Deactivation of Amberlite IR-120H catalyst during PAA formation (molar ratio of acetic acid to $H_2O_2 = 1:1$, catalyst loading = 471 mg/cm^3 , temperature = 40°C).

experiment, the used catalyst for the synthesis of PAA was washed with water thoroughly, subsequently dried in oven at 70°C for 48 h and reused for successive experiment. **Figure 7** depicts the evaluation of the activity of fresh and reused catalyst and it has been observed that the activity of the catalyst decreases with its usage. The concentration of PAA formed in first, second, and third cycles of catalyst was observed to be 3.375, 2.975 and 2.145 mol/L, respectively, at the end of 50 min of the experimental run. The possible reasons for the deactivation of the Amberlite IR-120H catalysts are neutralization of the sulfonic acid groups, catalyst shrinkage, or loss in pore sites [23–25]. In presence of ultrasonic irradiation, as all the catalyst particles remains suspended in the reaction media, the availability of active sites of catalysts can be considered as significantly enhanced. As a result of which the greater surface area would be available for reactions leading to enhanced reaction rates. Additionally, activation of the catalyst takes place in the presence of ultrasound as ultrasound acts as cleaning agent and adsorbed material gets cleaned by ultrasound. Therefore, the activity has not been decreased drastically with the use of catalyst.

3.5 Activity of Amberlite IR-120H catalyst in batch reactor

Figure 8 depicts the comparison of the activity of cation-exchange resin, that is, Amberlite IR-120H with H_2SO_4 homogeneous catalyst and ultrasound irradiation (40 kHz, 150 W) alone (in the presence of H_2O_2) in the synthesis of peracetic acid. These experiments were carried out under same catalyst loading, that is, 471 mg/cm^3 with molar ratio of acetic acid to $\text{H}_2\text{O}_2 = 1:1$ at temperature 40°C . It can be seen from the **Figure 8**, the concentration of PAA is 3.375 mol/L for cation-exchange resin, that is, Amberlite IR-120H at the end of 60 min for the same condition. Further, the PAA concentration was observed to be 2.55 mol/L at the end of 60 min for same experimental conditions at 1.1% H_2SO_4 catalyst loading. Without using the catalyst, the value PAA concentration obtained is 2.225 mol/L for same experimental conditions in the presence of H_2O_2 . Thus, it can be inferred that the concentration of PAA obtained is higher in case of cation-exchange resin, that is, Amberlite IR-120H in the presence of ultrasound. When only ultrasound is used, the concentration is 2.225 mol/L at the end of 60 min. It is attributed to the formation of H^\cdot and OH^\cdot radicals due to cavitation effects of ultrasound (Section 2.2), which accelerated the reaction rate leading to the formation of PAA.

3.6 Effect of ultrasound on PAA formation

To study the effect of ultrasound, three different sets of experiments were conducted: [1] without H_2O_2 in presence of ultrasound, [2] with H_2O_2 in presence of ultrasound, and [3] with H_2O_2 in absence of ultrasound at the same catalyst loading, that is, 471 mg/cm^3 with molar ratio of acetic acid to $\text{H}_2\text{O}_2 = 1:1$ at temperature 40°C . **Figure 9** depicts the effect of the cavitation effects of ultrasonic irradiations on the formation of H_2O_2 , that is, in turn on PAA formation. The maximum PAA concentration was observed to be 0.425 mol/L in the experiment carried out without H_2O_2 in presence of ultrasound. This is recognized to the H_2O_2 formation due to the cleavage of water molecules in the presence of ultrasonic irradiations as per the mechanism provided in the Section 2.2. The rupture of water molecules results in the formation of H^\cdot and OH^\cdot radicals, and combination two OH^\cdot radicals leads to the formation H_2O_2 [19], which is one of the reactant of the PAA formation reaction. This formed H_2O_2 that accelerates the PAA formation. In case of the experiments conducted in absence of ultrasound with H_2O_2 , the concentration of PAA was observed to be 2.00 mol/L , which is considerably higher than that of in the presence of ultrasound without H_2O_2 . Also, in the experiments conducted in presence

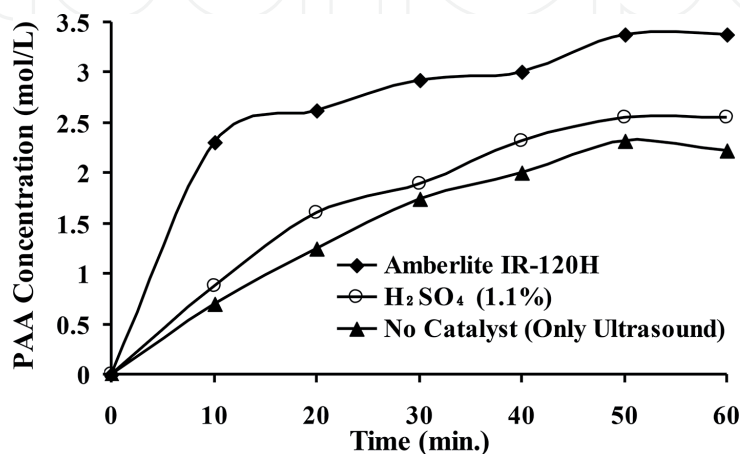


Figure 8. Comparative study on the activity of different catalyst during PAA formation (molar ratio of acetic acid to $\text{H}_2\text{O}_2 = 1:1$, catalyst loading of Amberlite IR-120H = 471 mg/cm^3 , temperature = 40°C).

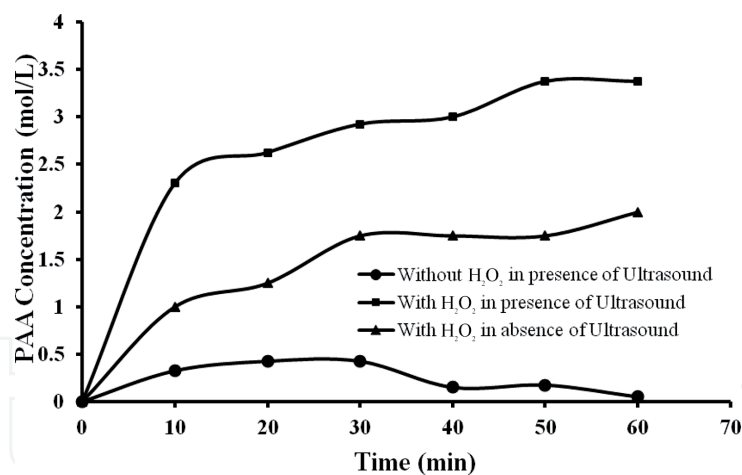


Figure 9. PAA formation in the presence of ultrasound without H₂O₂, in presence of ultrasound with H₂O₂ and in the absence of ultrasound with H₂O₂ (molar ratio of acetic acid to H₂O₂ = 1:1, catalyst loading = 471 mg/cm³, and temperature = 40°C).

of ultrasound with H₂O₂, the PAA concentration was observed to be significantly increased to 3.375 mol/L. This is due to the collective outcome of H₂O₂ added and generated in presence of ultrasound. Additionally, due to this surplus H₂O₂, the reaction becomes pseudo-first order and favors the PAA formation leading to suppression of reverse reaction and higher concentration of PAA in the final product.

4. Conclusions

The sonochemical synthesis of PAA in presence of Amberlite IR-120H as catalyst has been investigated. The optimum PAA concentration is obtained at temp. = 40°C, molar ratio of acetic acid to H₂O₂ 1:1, and at catalyst loading of 471 mg/cm³. Cavitation effects of ultrasonic irradiations play a significant role in the formation of PAA in batch reactor as it produces H₂O₂, which improves the reaction rate. Further, the use of batch reactor in the presence of ultrasonic irradiations enhances reaction rate significantly and reaction gets completed within merely 60 min compared to 30 h in batch reactor. The deactivation of Amberlite IR-120H catalyst occurs due to the neutralization of the sulfonic acid groups, catalyst shrinkage, or loss in pore sites. The experimental data were utilized for determination of activation energies for the synthesis and hydrolysis of PAA. The estimated activation energy for synthesis and for hydrolysis of PAA was found to be 40.6 and 22.43 kJ/mol, respectively. The activation energy for synthesis of PAA is significantly higher than the reported values in the literature. The observed effect can be attributed to the use of ultrasound during synthesis.

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