



Oxidation of Cyclohexanol and Cyclohexanone by Monochromate Ions in Organic Solvents and on Solvent Free Microwave Irradiation under Phase Transfer Catalysis - A Comparative Study

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Oxidation of cyclohexanol and cyclohexanone were carried out by acidified monochromate ions in ethyl acetate and toluene under phase transfer catalysis and also in solvent free condition under microwave irradiation. The extraction of monochromate ions from aqueous medium to organic phase was carried by employing various phase transfer catalysts in the presence of mineral acids. The effect of [catalyst] and [mineral acid] on extraction of monochromate from aqueous phase to organic phase was also studied. The product obtained, namely adipic acid obtained with both reactants was characterized by its melting point and infrared spectral technique. The reaction was over within 15 min with more than 85% yield at a temperature of 323 K under microwave irradiation where as it gave around 70% yield at 353 K within 150 min under phase transfer catalysis condition. The enhanced reaction rate and high yield of product substantiate the application of phase transfer catalytic technique under microwave irradiation for organic synthesis. A suitable mechanism for the oxidation of substrates by monochromate under phase transfer catalysis is also suggested.

Keywords: Oxidation, Extraction, Microwave irradiation, Monochromate ions, Phase transfer catalysis.

INTRODUCTION

Oxidation of organic substrates for the manufacturing of compounds like aldehydes, ketones, glycols, oximes, nitriles, azo compounds and carboxylic acids is having great industrial significance today [1,2]. The commonly used oxidants are permanganate, dichromate, hypochlorite, bromate, ferrate, etc. which are widely used in organic syntheses [3-6]. But these reactions are very slow and ineffective even at high temperature due to the inability of inorganic oxidants to dissolve in organic solvents where the substrate is dissolved. Rapid stirring at high temperature and the use of dipolar aprotic solvents like DMSO, DMF, etc. which can dissolve both organic substrate and inorganic oxidant were the earlier methods to conduct such reactions [7-9]. The applications of dipolar aprotic solvents have brought some improvements in such reactions. But disadvantages like the toxicity and high cost, requirement of very high reaction time and temperature, difficulty in product separation, lack of purity of products, etc. adversely affected this method for a wider applications. The introduction of phase transfer catalysis

(PTC) has revolutionized organic synthesis by overcoming all the difficulties faced in conventional methods [10-12], which involves the transfer of inorganic anions from aqueous medium into the non-polar organic medium in the form of an ion-pair with the cationic moiety of phase transfer catalyst having appropriate size and lipophilicity. The anions thus transferred and poorly solvated in the organic medium is found to exhibit greater reactivity. This enables the substrate to react faster in the non-polar medium in the presence of a phase transfer catalyst. The reaction can be accomplished in a biphasic system involving an inexpensive non-polar aprotic solvent and water without using highly polar solvents. The common phase transfer catalysts used are crown ethers and quaternary onium salts in which the latter is more suitable in selectivity, ease of use and of low cost [13-16]. Even a small quantity of phase transfer catalyst is able to transfer inorganic oxidant from the aqueous phase to organic phase in the form of an ion pair. The active oxidant as an ion pair in the organic phase interacts with the substrate and forms the product with enhanced rate at optimum conditions [17,18]. Cost reduction and pollution prevention are the

major aims of a chemical industry in the era of green chemistry today, which can be successfully accomplished by adopting a suitable PTC method [19-21].

There are reports on the synthesis of various organic compounds by oxidation reactions using permanganate, hypochlorite, bromate, ferrate, *etc.* under PTC conditions [22-25]. Potassium dichromate is a powerful oxidizing agent for the selective synthesis of various compounds in polar media and also used as an effective and selective oxidant in non-polar media under PTC conditions [26-29]. Starks & Liotta [30] had given a detailed account on chromate extraction, particularly, HCrO_4^- , CrO_4^{2-} , HCr_2O_7^- and $\text{Cr}_2\text{O}_7^{2-}$ into organic solvents using phase transfer catalysts. It was proved that under acidic conditions, HCrO_4^- and HCr_2O_7^- were readily phase transferred. But the transfer of chromate ions into organic phase will not occur in the absence of acid. According to the present study, the dichromate anion, $\text{Cr}_2\text{O}_7^{2-}$ is difficult to be phase transferred into the organic phase and is reported elsewhere [30]. This is due to the difficulty in transferring a bivalent anion. Hence acidified monochromate ion (HCrO_4^-) is the effective oxidant species which is transferred from aqueous to organic phase. A pinch of sodium carbonate is added to the acidified potassium dichromate solution in order to keep the pH of the solution in a range of 6-8. Under this pH, monochromate ions are formed predominantly and can be easily extracted to the organic phase.

The present study reports the oxidation of cyclohexanol and cyclohexanone under PTC conditions using monochromate ions as oxidant. Tetrabutylammonium bromide (TBAB), tetrabutylammonium chloride (TCMAC), tetrabutylammonium hydrogen sulfate (TBAHS) and cetyltrimethylammonium bromide (CTMAB) were used as phase transfer catalysts in ethyl acetate and in toluene under acidic condition. Extraction of monochromate ions from aqueous medium to ethyl acetate and toluene was carried out and the effect of [PTC] and $[\text{H}_2\text{SO}_4]$ on extraction was studied. The oxidation reaction was also carried out under solvent free condition with microwave irradiation. The combination of solvent free procedures and microwave irradiation can be used to carry out a wide range of reactions within short reaction time with high selectivity and yield [31-34].

EXPERIMENTAL

AnalaR grade potassium dichromate and sodium carbonate (E-Merck, India) were used as such and all the solutions were prepared in doubly distilled water. Cyclohexanol and cyclohexanone (Sisco Research Laboratories Pvt. Ltd., India) were of AnalaR grade and used after distillation under reduced pressure. The organic solvent ethyl acetate and toluene were purified according to the standard procedure [35,36]. The purified solvents were refluxed for 1-2 h with a mixture of phase transfer catalyst and potassium dichromate and then distilled. The phase transfer catalysts such as tetrabutylphosphonium bromide (TBPB), tetrabutyl ammonium hydrogen sulfate (TBAHS) (Merck KGaA, Germany) and tetrabutylammonium bromide (TBAB), cetyltrimethylammonium bromide (CTMAB) (Spectrochem India Ltd., Mumbai) were used as such.

General procedure: The extraction of monochromate ions from aqueous to organic medium with the aid of phase transfer catalysts was investigated and the effect of [PTC] and $[\text{H}^+]$ are reported. A potassium dichromate solution (20 mL, 0.01 mol dm^{-3}) containing $2 \text{ mol dm}^{-3} \text{ H}_2\text{SO}_4$ and a pinch of Na_2CO_3 were shaken with 20 mL ethyl acetate and toluene containing various [PTC]. The amount of HCrO_4^- extracted is determined by measuring its absorbance spectrophotometrically on Hitachi U-3010 UV-Visible spectrophotometer using 1 cm quartz cell at absorption maxima 365 nm. The effect of $[\text{H}^+]$ was studied by varying the concentration of added acid.

Synthetic analysis was carried out in a heterogeneous system. Cyclohexanol and cyclohexanone (0.1 mol) dissolved in 50 mL ethyl acetate and toluene in different bottles which contains 0.01 mol phase transfer catalyst were mixed with 50 mL HCrO_4^- extracted (0.5 mol). The solution mixture was refluxed for 2.5 h by maintaining at 80-85 °C. The unreacted monochromate ions were destroyed by the addition of sodium bisulfite. The solution was acidified with conc. HCl after 20 min. The solid products precipitated were collected by filtration and the non-aqueous layers were extracted with ether three times. The combined organic layer is then dried over anhydrous magnesium sulphate and concentrated by rotary evaporator. The products obtained were dissolved in 5% NaOH solution and combined with basic solutions from previous extractions. These solutions were acidified with conc. HCl and extracted with ether three times. Then they were dried over magnesium sulphate and evaporated to give the purified products. The products obtained were analyzed by melting point determination and infrared spectral technique.

The reaction was also carried under solvent free condition by microwave irradiation. The reaction mixture containing substrates (0.02 mol) in 20 mL ethyl acetate and toluene containing 0.005 mol phase transfer catalyst were mixed with 10 mL $\text{K}_2\text{Cr}_2\text{O}_7$ (0.1 mol) containing $2 \text{ mol dm}^{-3} \text{ H}_2\text{SO}_4$ and a pinch of Na_2CO_3 . These reaction mixtures in separate bottles were heated in a microwave oven for about 20 min. The solutions were taken out and acidified with conc. HCl. The solid product precipitated was extracted and isolated as explained above and was analyzed by melting point determination and infrared spectral technique.

RESULTS AND DISCUSSION

The results on the extraction of monochromate ions from aqueous phase to organic phase with the aid of phase transfer catalyst are presented in Table-1. All the phase transfer catalysts were found to be effective in bringing out monochromate ions as ion pair from aqueous phase to organic phase but the extent of extraction varies slightly in the order $\text{TCMAC} > \text{TBAB} > \text{TBAHS} > \text{CTMAB}$. This can be explained on account of changes in combination of alkyl or aryl groups and also due to the difference in the activity of anions for phase transfer. The major requirement of the cationic part of a phase transfer catalyst is that it collectively should have sufficient organic structure to transfer the desired anion into the organic phase. It is already known that larger alkyl or aryl groups with almost symmetric

TABLE-1
EFFECT OF [PT CATALYST] ON EXTRACTION OF MONOCHROMATE IONS
[K₂Cr₂O₇] × 10² = 1.0 mol dm⁻³ [H₂SO₄] = 2.0 mol dm⁻³; Temperature = 300 K; Solvent = Ethyl acetate and toluene

| [PT] catalyst × 10 ³ (mol dm ⁻³) | Time (min) | % of HCrO ₄ ⁻ extracted into ethyl acetate | | | | % of HCrO ₄ ⁻ extracted into toluene | | | |
|---|------------|--|------|-------|-------|--|------|-------|-------|
| | | TCMAC | TBAB | TBAHS | CTMAB | TCMAC | TBAB | TBAHS | CTMAB |
| 5 | 15 | 51.2 | 49.4 | 38.4 | 34.6 | 48.4 | 44.2 | 34.9 | 33.8 |
| | 30 | 54.6 | 51.8 | 41.6 | 38.7 | 52.1 | 46.6 | 39.8 | 38.5 |
| | 45 | 58.5 | 54.2 | 43.8 | 40.2 | 54.7 | 51.2 | 41.4 | 40.5 |
| | 60 | 61.8 | 58.4 | 47.4 | 44.6 | 57.3 | 54.7 | 44.6 | 43.9 |
| 10 | 15 | 61.6 | 59.6 | 54.8 | 48.9 | 58.2 | 57.5 | 50.6 | 44.4 |
| | 30 | 64.8 | 60.2 | 56.4 | 52.3 | 61.6 | 59.9 | 53.5 | 47.2 |
| | 45 | 70.8 | 66.4 | 60.2 | 56.7 | 63.6 | 61.2 | 58.4 | 49.9 |
| | 60 | 75.6 | 69.8 | 64.6 | 60.3 | 70.5 | 64.7 | 60.6 | 52.2 |
| 20 | 15 | 83.2 | 81.6 | 76.4 | 64.6 | 72.2 | 70.7 | 64.4 | 54.6 |
| | 30 | 86.8 | 82.4 | 76.6 | 69.6 | 74.8 | 73.6 | 67.8 | 57.8 |
| | 45 | 88.8 | 82.6 | 78.2 | 72.4 | 80.2 | 77.8 | 70.7 | 63.3 |
| | 60 | 88.8 | 82.8 | 78.2 | 74.5 | 84.4 | 77.8 | 72.4 | 67.7 |
| 30 | 15 | 88.8 | 82.6 | 78.2 | 74.8 | 84.2 | 77.8 | 72.6 | 71.5 |
| | 30 | 89 | 82.8 | 78.4 | 74.6 | 84.4 | 78.3 | 73.4 | 71.8 |
| | 45 | 89.2 | 82.8 | 78.8 | 74.6 | 84.6 | 78.2 | 73.4 | 71.8 |
| | 60 | 89.2 | 82.6 | 78.8 | 74.8 | 84.6 | 78.3 | 73.6 | 72.0 |

structure are most effective both in anion transfer and in reactivity. The activity of a quaternary salt as a phase transfer catalyst also depends on the anion originally present. The quaternary salts are useful as phase transfer catalysts only when the anion accompanying the catalyst is distributed in the organic phase to a much lesser extent than the anion to be reacted. In general, the large, lipophilic quaternary cations are soft in the hard and soft acids and bases (HSAB) concept and so that this cation tends to pair with the softest anions available in the solution and transfer it into the organic phase [35]. Therefore, TCMAC with more bulky and symmetrical alkyl groups has higher reactivity for extraction on comparison with other phase transfer catalyst used. The other phase transfer catalysts, TBAB, TBAHS and CTMAB are having almost similar cationic moiety and their extraction capacity is almost similar in ethyl acetate. The percentage of extraction is more in ethyl acetate than in toluene due to the higher polarity of ethyl acetate. The solubility and partitioning behaviour of the quaternary salts are significantly affected by slight changes in the nature of organic phase. Solubility and partitioning of quaternary salts are increased by increasing the polarity of the aprotic organic phase.

The influence of concentration of mineral acids on extraction is carried out and found that the extraction of monochromate is not possible in the absence of mineral acids (Table-2). The extraction percentage increases with increase in [mineral

acid] up to 2 mol dm⁻³ and then remains constant on increasing it.

Oxidation of cyclohexanol and cyclohexanone by phase transfer catalyzed monochromate ions gave adipic acid as the product with a yield of > 70%. The obtained products were analyzed by melting point in triplicate and was found to be 151 ± 2 °C, which was in accordance with reported value of 153 °C. The infrared absorption spectra of the obtained products in both oxidation reactions were recorded from KBr pellets using Jasco FT-IR 4100 spectrophotometer (Japan) and is shown in Fig. 1.

The infrared absorption spectra of the obtained products on the oxidation of cyclohexanol and cyclohexanone by phase transferred monochromate ions showed a broad peak at 2960 cm⁻¹ (O-H *str.*), 2850 cm⁻¹ (C-H *str.* superimposed upon O-H *str.*), 1710 cm⁻¹ (C=O *str.*), 1410 cm⁻¹ (C-O-H in-plane band), 1220 cm⁻¹ (C-O *str.*) and 920 cm⁻¹ (O-H out-of-plane bending).

Presence of peaks explained leads to the conclusion that the product obtained on chromate oxidation of cyclohexanol and cyclohexanone is adipic acid which was further confirmed on comparison with the infrared spectrum of adipic acid available in literature. The oxidation of cyclohexanol and cyclohexanone was also carried out under solvent free conditions on microwave irradiation and the results are compared with that of phase transfer catalysis assisted oxidation.

TABLE-2
EFFECT OF [MINERAL ACID] ON EXTRACTION OF MONOCHROMATE IONS
[K₂Cr₂O₇] × 10² = 1.0 mol dm⁻³, [PT catalyst] × 10³ = 20 mol dm⁻³, Temperature = 300 K Solvent = Ethyl acetate and toluene

| [H ₂ SO ₄] (mol dm ⁻³) | Time (min) | % of HCrO ₄ ⁻ extracted into ethyl acetate | | | | % of HCrO ₄ ⁻ extracted into toluene | | | |
|---|------------|--|------|-------|-------|--|------|-------|-------|
| | | TCMAC | TBAB | TBAHS | CTMAB | TCMAC | TBAB | TBAHS | CTMAB |
| 0 | 15 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | 30 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 1 | 15 | 80.5 | 78.8 | 74.8 | 64.2 | 69.8 | 66.6 | 62.2 | 52.6 |
| | 30 | 81.2 | 78.8 | 74.8 | 64.4 | 71.4 | 68.9 | 63.4 | 53.5 |
| 2 | 15 | 83.2 | 81.6 | 76.4 | 64.6 | 72.2 | 70.7 | 64.4 | 54.6 |
| | 30 | 86.8 | 82.4 | 76.6 | 69.6 | 74.8 | 73.6 | 67.8 | 57.8 |
| 3 | 15 | 89.2 | 82.6 | 76.6 | 74.6 | 84.6 | 78.2 | 73.4 | 71.8 |
| | 30 | 89.2 | 82.8 | 76.6 | 74.8 | 84.6 | 78.3 | 73.6 | 72.0 |

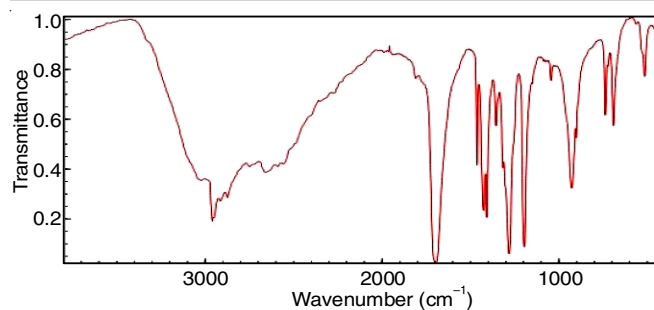
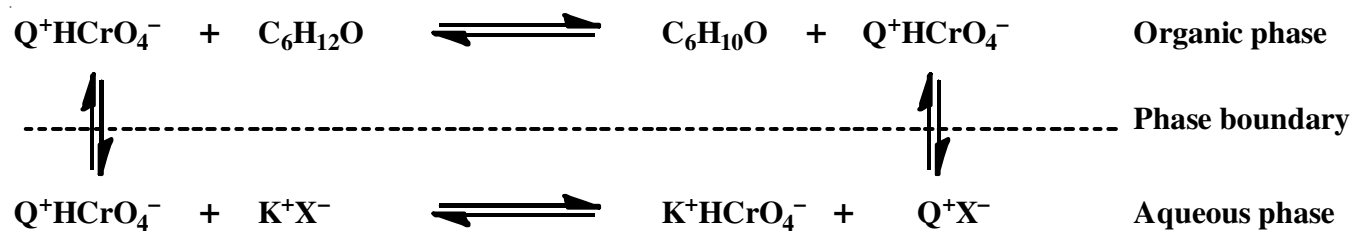
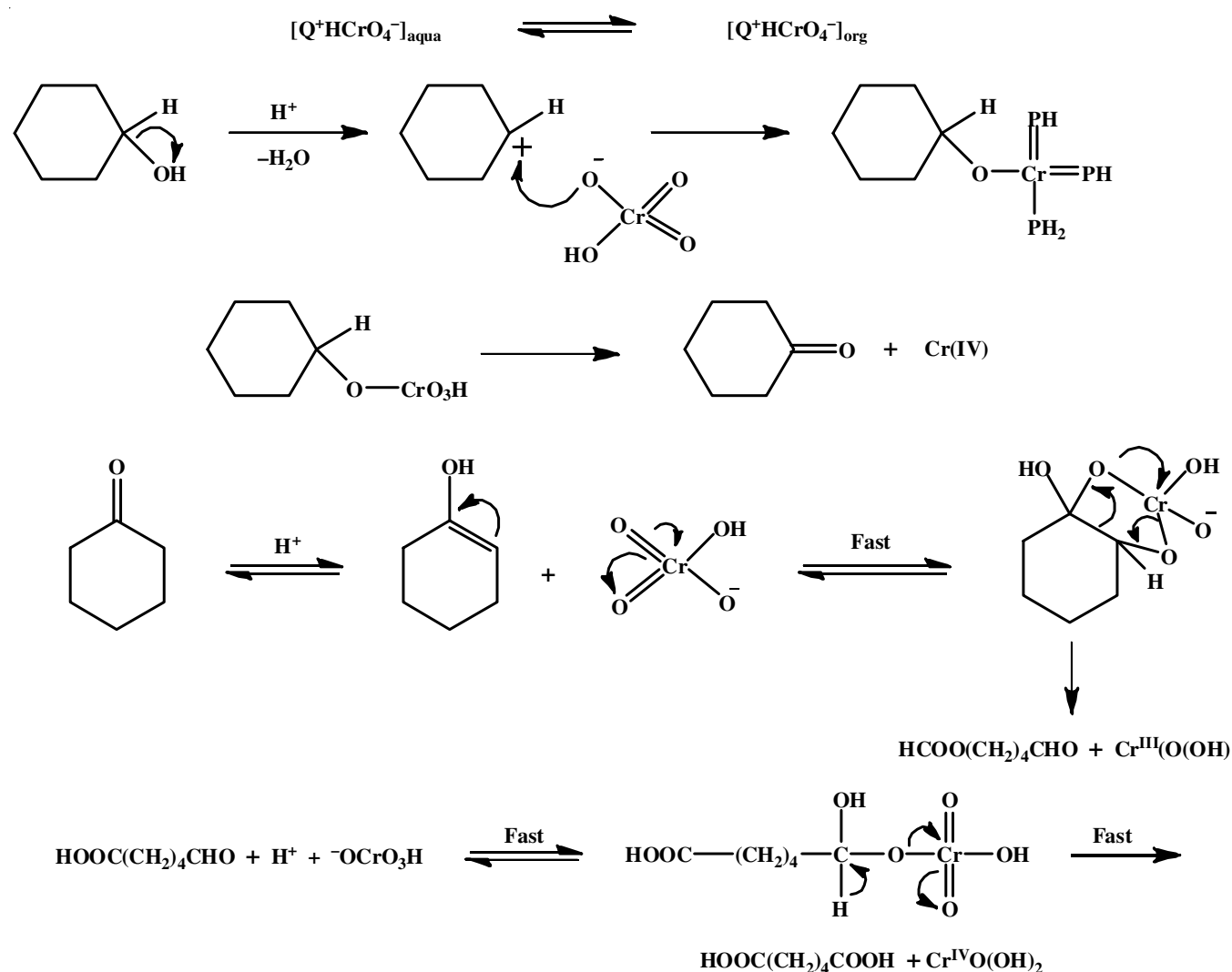


Fig. 1. Infrared spectrum of product on chromate oxidation of cyclohexanol and cyclohexanone

From Table-3, it is very clear that the product yield is significantly increased in oxidation reaction under microwave irradiation and also the reaction time and temperature are considerably decreased. The yield is increased more than 30% and reaction time is reduced from 3.5 h to just 15 min whereas the reaction temperature is decreased from 85 to 50 °C. Phase transfer catalysis (PTC) method is universally proved as a superior technique to overcome all the difficulties in conventional methods like rapid stirring at high temperature and use of dipolar aprotic solvents in industrial synthesis of various organic compounds with high yield under optimum conditions. Microwave



Scheme-I: Stark's phase transfer catalytic cycle for the monochromate oxidation of cyclohexanol



Scheme-II: Mechanistic path for the PT catalyzed oxidation of cyclohexanol and cyclohexanone by phase transferred monochromate

TABLE-3
COMPARISON OF OXIDATION REACTIONS UNDER PTC AND MICROWAVE IRRADIATION

| Substrate | PTC reaction | | | Microwave irradiation reaction | | |
|---------------|--------------|------------------|-----------|--------------------------------|------------------|-----------|
| | Time (min) | Temperature (°C) | Yield (%) | Time (min) | Temperature (°C) | Yield (%) |
| Cyclohexanol | 150 | 80-85 | 73 | 15 | 50 | 90.3 |
| Cyclohexanone | 150 | 80-85 | 70.6 | 15 | 50 | 89.3 |

assisted organic synthesis promises advantages like eco-friendliness like omission of toxic solvents, high reaction rate, high yield and considerable reduction in reaction time. All these merits make microwave assisted reactions as a green method for the synthesis of various industrially important chemicals. But applications of microwave irradiation method to large scale synthesis in chemical industries is still a problem, which yet to be solved by scientific community.

A suitable reaction path called Stark's phase transfer catalytic cycle for the transfer of monochromate ion from aqueous phase to organic phase as an ion pair and subsequent reaction in organic phase is given in **Scheme-I**. A plausible mechanism for the oxidation of cyclohexanol to cyclohexanone by acidified monochromate in ethyl acetate and subsequent oxidation to adipic acid is given in **Scheme-II**.

Conclusion

The present work reports a comparative study on the monochromate oxidation of cyclohexanol and cyclohexanone in organic solvent under phase transfer catalysis (PTC) condition and solvent free microwave irradiation condition. The extraction of monochromate ions from aqueous medium to organic phase was carried out and the effect of [PTC] and [H₂SO₄] on extraction was also reported. The reaction was found to be very fast at optimum conditions with high yield under microwave irradiation on comparison with PTC condition. Even though microwave irradiation method is found to be more effective than PTC method, lack of application of it for large scale chemical synthesis makes PTC method more suitable in chemical industry today.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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