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Research Paper

EFFICIENCY OF AN IONIC LIQUID AS CATALYST FOR DEGRADATION OF WASTE NYLON-6 AND ITS OPTIMIZATION STUDY

Sunil R. Chikte[1], Swapnil V. Madhamshettiwar[2]

[1,2] *Assistant Professor, Department of Chemistry, Sardar Patel Mahavidyalaya, Chandrapur 442401 (M.S.) India*

Corresponding author: sunilchikte78@gmail.com

Abstract:

With the increasing rate of production and usage ofnylon-6, the environmental effect of plastics is currently a global concern. The widespread and careless use and disposal of these non-biodegradable materials has severely disrupted and harmed the environment and its biodiversity. To overcome these problems, ionic liquid has shown the efficiency as catalyst for the hydrolysis of waste nylon-6 into N-benzoyl-Ɛ-caprolactam. Ionic liquids [emim]BF_4, [bmim]BF_4, and [bmim]PF_6 has successfully hydrolyzed the waste nylon-6 at different extent. Maximum yield, 4.38g of desired monomer, was obtained when hydrolysis was carried in the presence of .9 g (.1mol) of [emim]BF_4 as a catalyst at 120 ^{0}C for five hours. Hydrolysis in the presence of IL [emim]BF_4 as a catalyst showed the maximum breakdown of nylon-6 in a shorter time than ILs [bmim]BF_4 and [bmim]PF_6. The hydrophilic nature of IL [emim]BF_4 and [bmim]BF_4 has shown an efficient tendency to catalyze the hydrolysis of nylon-6, while the hydrophobic nature of [bmim]PF_6 has the least tendency to catalyze the hydrolysis of nylon-6. The product was characterized by FT-IR, ^{1}H, and ^{13}C NMR techniques.

Key words: Catalyst, Ionic liquids, Waste nylon-6, N-benzoyl- Ɛ-caprolactam, Hydrophilic, Hydrophobic

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INTRODUCTION

Plastics are a necessary component of contemporary life. Since the 1950s, when plastics were first produced in large quantities, it has become nearly impossible to live without them [1]. Due to their molecular makeup and additions, plastics are incredibly adaptable materials that have significantly improved human welfare through a various useful applications. However, due to their relative inertness and lack of biodegradability, plastics pose a significant treatment and disposal challenge in the management of urban solid waste [2]. Unquestionably, these non-biodegradable materials' widespread and careless use and disposal have severely disrupted and harmed the environment and its biodiversity [3]. Due to the restricted means of treatment and disposal combined with the increasing production and usage rates, plastics' environmental effect is currently a global concern. Polyamide materials are commonly used because of their outstanding performance. Nylon-6 and nylon-66 comprises approximately 90% of all amide products, and both are equally significant. Consequently, research on polyamides primarily concentrates on these two types, and this study also addresses them. The increasing production rate of nylon-6 and nylon-66 is causing environmental pollution. Various studies have shown that there is an urgent need to solve the environmental pollution caused by these polyamides 4-6. When polyamides are

disposed of in landfills, they can persist for several decades or even centuries 7. This process produces toxic and dangerous gases that cause air pollution 8. Furthermore, many unacceptable disposal practices are frequently used in nations like India, including open dumping, uncontrolled incineration 9, unscientific composting and incorrect landfilling 10. Growing public concern over this issue has sparked interest in the study of the biodegradation of naturally very stable polymers, such as polyethene and polystyrene. Only 10% of the 140 million tons of synthetic polymers generated annually worldwide are recycled or reused due to the scarcity of disposal options 11. To overcome this problem, various methods have been developed to dispose of the waste nylon, and the degradation of waste nylon into its useful monomers was found to be the best method to solve these issues. Any physical or chemical change in a polymer's properties brought on by outside factors (such as light, heat, moisture, etc.), chemical reactions, or biological activity is known as polymer degradation. Chemical processes such as pyrolysis 12-13, solvent precipitation theory 14-15, alkaline hydrolysis 16, acidic hydrolysis 17-18, hydrothermal reaction 19, microwave assisted hydrolysis 20, ammonolysis 21, alcoholysis 22, enzymatic hydrolysis 23 and, environmental degradation 24-25 are used to break down waste polymers. Some of the methods mentioned above use hazardous and volatile reagents, which not only pollute the environment but also involve harsh reaction conditions. The need to substitute these hazardous volatile solvents has become urgent, as many of them are flammable, toxic, and quite dangerous compared to ionic liquids (ILs). This issue has gained significant attention in both academia and industry, primarily due to the growing emphasis on adopting cleaner technologies as a global priority.

ILs is gaining attention as potential replacements for volatile organic solvents. They are categorized as green solvents since they are recyclable, nonflammable, and non-volatile. They are regarded as favorable medium candidates for chemical syntheses because of their exceptional qualities, including their excellent solvating potential 26, thermal stability 27, and their adjustable properties by appropriate cations and anions 28. Ionic liquids have been demonstrated during the last two decades to be extremely promising "green" solvents that provide several benefits over conventional organic solvents 29-30. The chemical industry regards them as valuable solvents because of their remarkable solvent properties, stability at high temperatures, and lack of volatility. Ionic liquids (ILs) have a melting point less than 100 °C or even room temperature (RTILs), since they only include organic cations and inorganic or organic anions 31. Interest in ionic liquids has grown significantly as a potential replacement for volatile organic solvents. ILs is also called greener solvents because of their recyclable, nonflammable, and non-volatile properties.

Kamimura A. et al. (2007) proposed using ionic liquids to depolymerize polyamides, suggesting that the counter anions in these ionic liquids play a crucial role. It was found that, after six hours of reaction at 300 °C, the yield of caprolactam could reach 86% when using DMAP (4-dimethylaminopyridine) as a catalyst and N-methylpyrrolidone as a solvent 32.

Ionic liquid [Bmim][FeCl4] can be used to catalyze the depolymerization of lignin to methyl p-hydroxycinnamate. Overall catalyst [FeCl4]− found an important part during the catalytic process, having two activation modes and three distinct reaction routes that can be used to achieve the complete depolymerization of the model compound lignin 33. Ionic liquid [emim]BF4 is an example of a hydrophilic ionic that effectively degraded nylon-6 into caprolactam monomer. The ionic liquid was successfully separated from the obtained monomer by the simple extraction method 34. Without the use of conventional acid or base catalysts, the methanolysis of poly (lactic acid) (PLA) was accomplished with ease at lower pressures and temperatures when the ionic liquid [Bmim][Ac] was used as a catalyst 35. There has been a considerable amount of research on the breakdown of PET, polyester, lignin, cellulose, etc., but more work is needed on the breakdown of polyamide nylon-66.

This research aims to identify ionic liquids as catalysts for the hydrolysis of waste nylon-66 to produce monomers. Specifically, it examines 1-ethyl-3-methylimidazolium tetrafluoroborate ([emim]BF4), 1-butyl-3-methylimidazolium tetrafluoroborate ([bmim]BF4), and 1-butyl-3-methylimidazolium hexafluorophosphate ([bmim]PF6). The study will compare the efficiency of these ionic liquids and optimize nylon-66 degradation at various temperatures and times, measuring the yield of dibenzoyl derivatives of hexamethylenediamine (DBHMD) and adipic acid.

Material and experimental method

Material

All the different types of ILs used in the present study were prepared in the laboratory. The 1-methylimidazole (99 % pure) required for the synthesis of ILs was obtained from Sigma-Aldrich all other chemical were of analytical grade. 5N HCl and 5N NaOH solutions were prepared by using double distilled water.

Experimental method

Collected Waste nylon-66 was contaminated with dust particles and some other microorganisms. To purify waste nylon-66, it was treated with a 1 g/L solution of nonionic detergent at around 70–80 °C for 6-7 hours. Furthermore, to remove any surface impurities, it was properly cleaned with distilled water and then allowed to dry at around 60–70 °C for 3–4 hours.

0.2 g of nylon-6 and 20 ml of 5N HCl were refluxed in the presence of 1.9 g (0.1 mol) of [emim]BF4 as a catalyst for 5 hours at a temperature range of 80-120 °C. After cooling the reaction mixture, the unreacted nylon-6 was separated using simple filtration. Following filtration, the reaction mixture was neutralized with a slight excess of 5N NaOH to achieve a slightly alkaline pH (7-7.5). This was followed by benzoylation, during which benzoyl chloride (C_6H_5COCl) was added to the alkaline solution. As a result, N-benzoyl ε-caprolactam (the benzoyl derivative of ε-caprolactam) precipitated out. The product was washed with water and recrystallized using ethanol.

The same procedure was repeated with different concentration of IL [emim]BF4 at different time and temperature.

Similarly the same procedure was performed for the 0.05 mol, 0.075 mol, and 0.1 mol of IL [bmim]BF4 and [bmim]PF6 by varying the reaction parameters like time and temperature for optimization study.

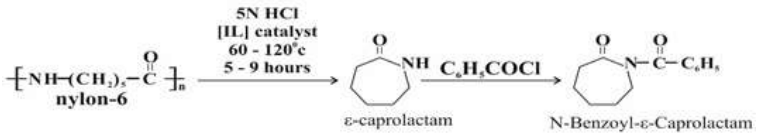

Figure1: Hydrolysis of nylon-6 using IL as catalyst

Result and discussion

Hydrolysis of nylon-6 using [emim]BF4 as catalyst

The hydrolysis of nylon-6 was performed using different concentrations of [emim]BF4 and at various times and temperatures. The results are summarized in the following table.

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Table 1: Hydrolysis of nylon-6 using [emim]BF4 as catalyst

0.95 g (0.05 mol.) of [emim]BF4				
Sr. no.	**Conc. of [emim]BF4 (g)**	**Time (hrs.)**	**Temp. (^{0}C)**	**N-benzoyl Ɛ-caprolactam (g)**
Different temp.				
1	0	5	60	1.09
2	.095	5	60	1.23
3	0.95	5	80	1.93
4	0.95	5	100	2.42
5	0.95	5	120	2.97
6	0.95	5	140	2.67
7	0	5	120	1.73
Different time				
8	0.95	4	120	2.22
9	0.95	3	120	1.89
1.425 g (0.075 mol.) of [emim]BF4				
Different temp.				
10	1.425	5	60	1.52
11	1.425	5	80	2.32
12	1.425	5	100	2.76
13	1.425	5	120	3.62
14	1.425	5	140	2.90
Different time				
15	1.425	4	120	3.20
16	1.425	3	120	2.61
1.9 g (0.1 mol.) of [emim]BF4				
Different temp.				
17	1.9	5	60	1.93
18	1.9	5	80	2.62
19	1.9	5	100	3.11
20	1.9	5	120	4.38
21	1.9	5	140	3.68
Different time				
22	1.9	4	120	3.31
23	1.9	3	120	2.93

At first, the reaction was run for five hours at 60 ^{0}C and 120 ^{0}C in the absence of IL catalyst. The resultant yield of the N-benzoyl Ɛ-caprolactam was found to be 1.09 and 1.73 g, respectively. But at 120 ^{0}C, the presence of IL as catalyst produced a larger monomer. From the above results, it was observed that as the concentration of catalyst, time and temperature increases, the degradation process also increases. Maximum yield, 4.38g of desired monomer, was obtained when hydrolysis was carried in the presence of 1.9 g (.1mol) of [emim]BF4 as a catalyst at 120 ^{0}C for five hours.

When the temperature increased to 140 ^{0}C, the monomer yield was decreased to 3.68 g.

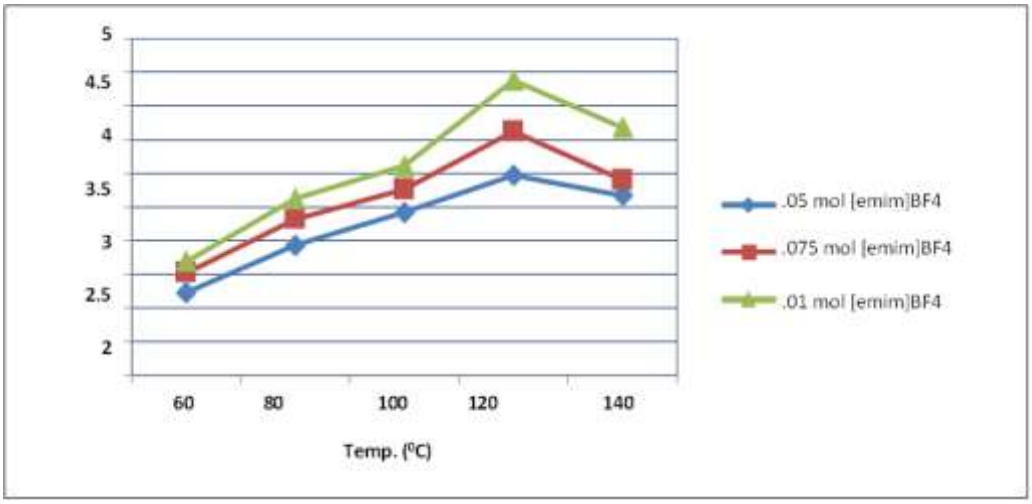

Figure 2: N-Benzoyl Ɛ- Caprolactam in different conc. of IL [emim]BF4 at different temp

Hydrolysis of nylon-6 using [bmim]BF4 as catalyst

Below are the results of the hydrolysis of nylon-6 at different concentrations of IL [bmim]BF4 as a catalyst at different times and temperatures.

Table 2: Hydrolysis of nylon-6 using [bmim]BF4 as catalyst

1.15 g (0.05 mol.) of [bmim]BF4				
Sr. no.	**Conc. of [emim]BF4 (g)**	**Time (hrs.)**	**Temp. (^{0}C)**	**N-benzoyl Ɛ-caprolactam (g)**
Different temp.				
1	0	7	60	1.13
2	1.15	7	60	1.39
3	1.15	7	80	1.62
4	1.15	7	100	2.01
5	1.15	7	120	2.43
6	1.15	7	140	2.27
7	0	7	120	1.92
Different time				
8	1.15	6	120	2.22
9	1.15	5	120	1.89
1.725 g (0.075 mol.) of [bmim]BF4				
Different temp.				
10	1.725	7	60	1.52
11	1.725	7	80	1.98
12	1.725	7	100	2.03
13	1.725	7	120	2.95
14	1.725	7	140	2.63
Different time				

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15	1.725	6	120	3.20
16	1.725	5	120	2.61
2.3 g (0.1 mol.) of [bmim]BF4				
Different temp.				
17	2.3	7	60	1.82
18	2.3	7	80	2.12
19	2.3	7	100	2.89
20	2.3	7	120	3.73
21	2.3	7	140	3.31
Different time				
22	2.3	6	120	3.03
23	2.3	5	120	2.41

The degradation of nylon-6 using .1 mol of IL [bmim]BF4 as a catalyst yields a lesser amount of N-benzoyl Ɛ-caprolactam than .1 mol of IL [emim]BF4. This is particularly significant when the reaction is performed at 120 0C for five hours, as it indicates that IL [bmim]BF4 is less efficient in catalyzing the hydrolysis of nylon-6 than IL [emim]BF4. However, the yield of monomer was still less than in the case of IL [emim]BF4, possibly due to the high viscosity of [bmim]BF4.

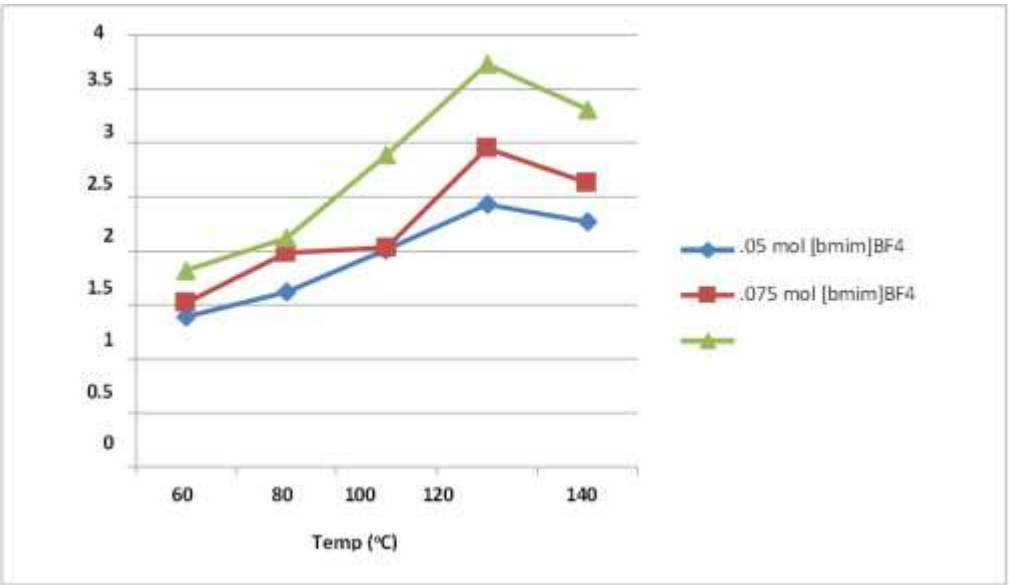

Figure 3: N-Benzoyl Ɛ- Caprolactam in different conc. of IL [bmim]BF4 at different temp.

Hydrolysis of nylon-6 using [bmim]PF6 as catalyst

Below are mentioned the degradation of nylon-6 at different times, temperatures, and concentrations of IL [bmim]PF6 as a catalyst.

Table 3: Hydrolysis of nylon-6 using [bmim]PF_6 as catalyst

Sr.no.	Conc. of [emim]BF_4 (g)	Time (hrs.)	Temp. (^{0}C)	N-benzoyl Ɛ-caprolactam (g)
1.4 g (0.05 mol.) of [bmim]PF_6				
Different temp.				
1	0	9	60	1.19
2	1.4	9	60	1.33
3	1.4	9	80	1.42
4	1.4	9	100	1.58
5	1.4	9	120	2.20
6	1.4	9	140	2.09
7	0	9	120	1.89
Different time				
8	1.4	8	120	2.10
9	1.4	7	120	1.89
2.1 g (0.075 mol.) of [bmim]PF_6				
Different temp.				
10	2.1	9	60	1.38
11	2.1	9	80	1.51
12	2.1	9	100	1.76
13	2.1	9	120	2.39
14	2.1	9	140	2.23
Different time				
15	2.1	8	120	1.81
16	2.1	7	120	1.64
2.8 g (0.1 mol.) of [bmim]PF_6				
Different temp.				
17	2.8	9	60	1.46
18	2.8	9	80	1.73
19	2.8	9	100	1.98
20	2.8	9	120	2.41
21	2.8	9	140	2.29
Different time				
22	2.8	8	120	2.03
23	2.8	7	120	1.84

.1 mol of [bmim]PF_6 as a catalyst in the hydrolysis of nylon-6 gave less amount of N-benzoyl Ɛ-caprolactam than .1 mol of [bmim] BF_4 at 120 ^{0}C when the reaction was run for seven hours. The hydrolysis of nylon-6 in the presence of .1mol of [bmim]BF_4 as a catalyst at 120 ^{0}C for seven hours yields a monomer equal to 3.73 g whereas .1 mol of [bmim]PF_6 as a catalyst at the same temperature and time yields only 1.84 g.

The maximum yield of N-benzoyl Ɛ-caprolactam was about 2.41g, when the reaction was performed for

nine hours at a temperature of 120 ^{0}C. This was achieved in the presence of 2.8 g (.1 mol.) of IL [bmim]PF6, a catalyst that played a crucial role in the reaction. The difference in yield in the presence (2.41g) and absence (1.89 g) of IL as a catalyst was minimal. Even when using IL [bmim]PF6, the yield of N-benzoyl Ɛ-caprolactam was lower, despite the nine-hour reaction time. IL [bmim]PF6 demonstrated lower efficiency in catalyzing the hydrolysis of nylon-6. This can be attributed to the water immiscibility of [bmim]PF6 and the presence of a larger alkyl group (non-polar) and PF_6^- anion, which reduces its ability to diffuse water into the nylon-6 structure. As a result, [bmim]PF6 has the least tendency to catalyze the degradation of nylon-6 in the hydrolysis method.

Characterization of monomers

^{1}H NMR of N-benzoyl Ɛ-caprolactam

^{1}H NMR (δ ppm) (500 MHz, CDCl3): N-benzoyl-Ɛ-caprolactam contains aromatic ring protons that exhibit multiplets in the range of 7.42 to 8.12 ppm. In contrast, the protons of the Ɛ-caprolactam ring show peaks between 1.34 and 4.39 ppm. Specifically, the multiplet at 7.42 to 8.12 ppm correspond to five hydrogen atoms from the aromatic ring, while the peaks from 1.34 to 4.39 ppm correspond to five >CH2 groups in the caprolactam ring. Additionally, the CDCl3 solvent displays a peak at 7.55 ppm

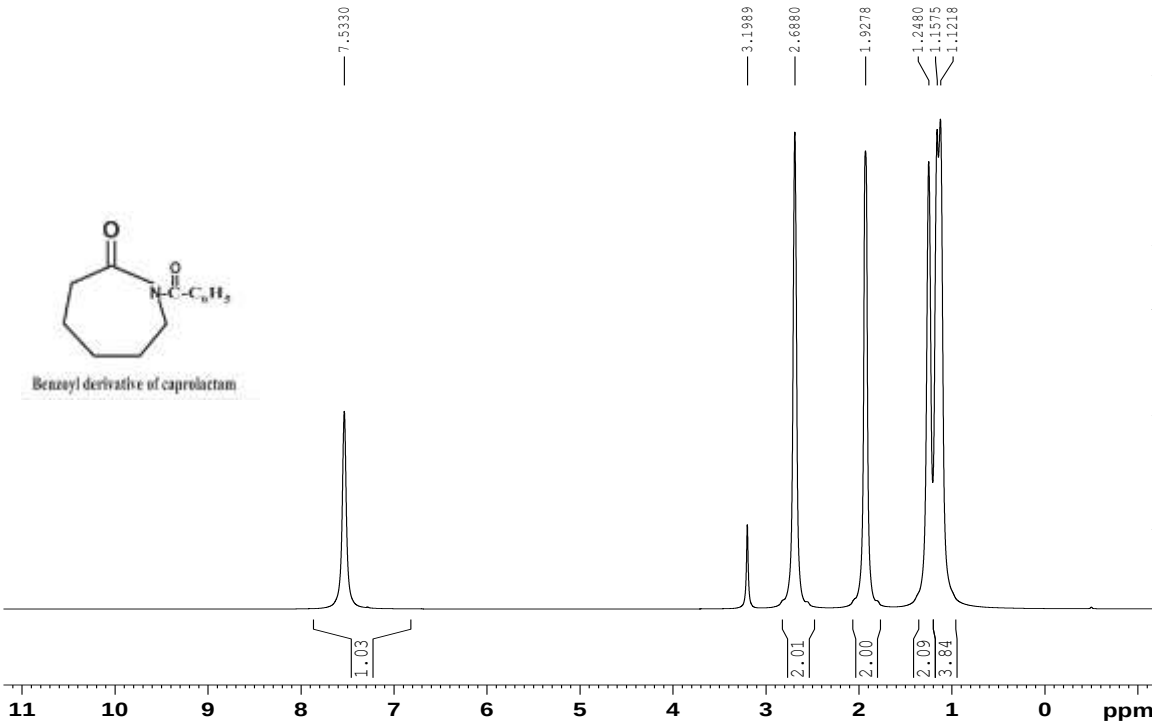

Figure 4: ^{1}H-NMR of Ɛ-caprolactam

^{13}C NMR (δ ppm) (500 MHz, $CDCl_3$): N-benzoyl-Ɛ-caprolactam exhibits a peak at 172.45 ppm, which is attributed to the carbonyl (>C=O) group. The carbon atoms within the caprolactam ring display peaks ranging from 14.09 to 61.72 ppm. A triplet at 77.11 ppm corresponds to the $CDCl_3$ solvent.

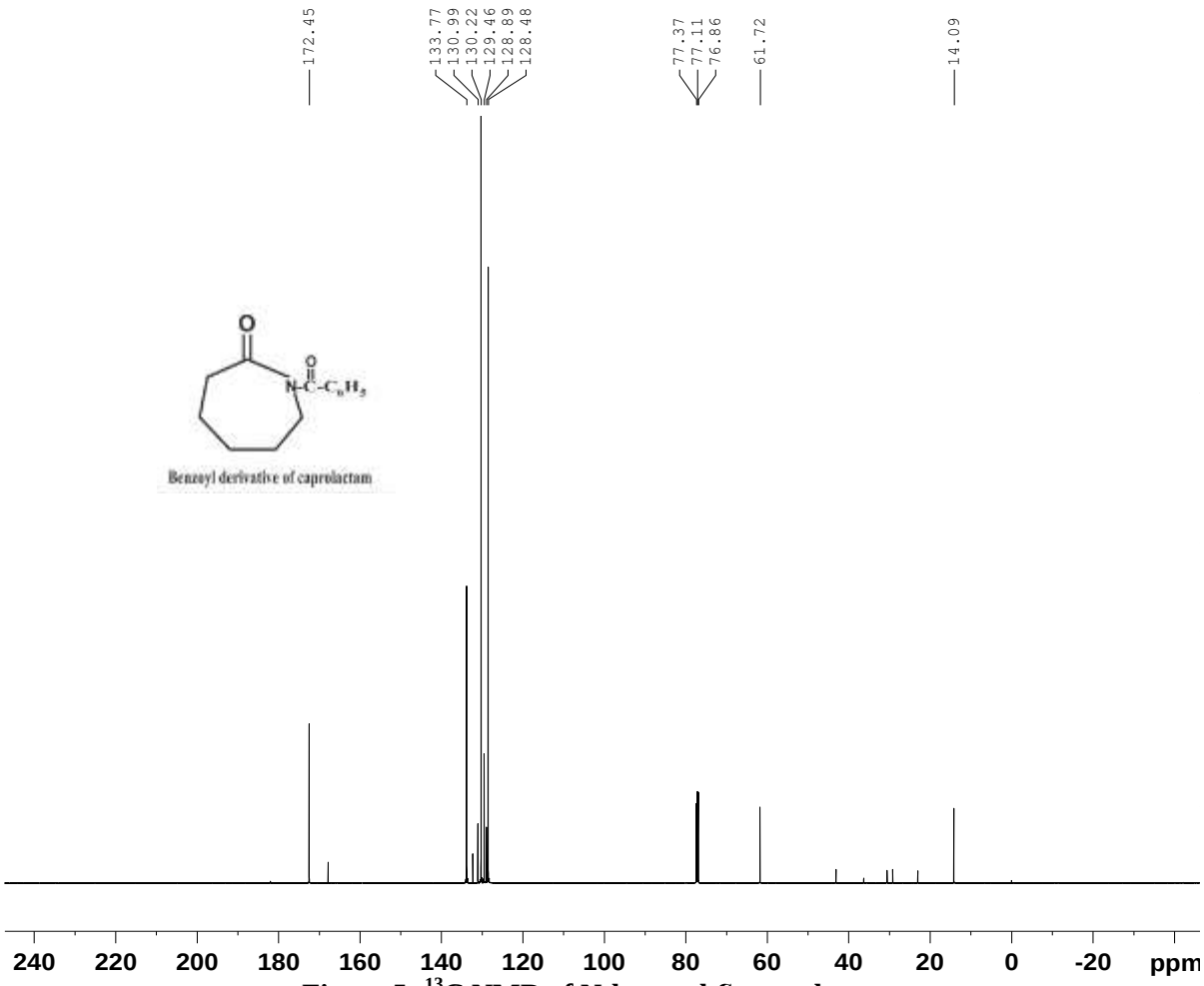

Figure 5: ^{13}C NMR of N-benzoyl-Ɛ-caprolactam

FT-IR (cm^{-1}): 2835-3071 (C-H str.), 1686 (C=O str.), 1600-1400 (C=C str. in aromatic ring). The bands below 1500 cm^{-1}(fingerprint region) are due to C-O, C-N, and C-C stretching and bending vibrations.

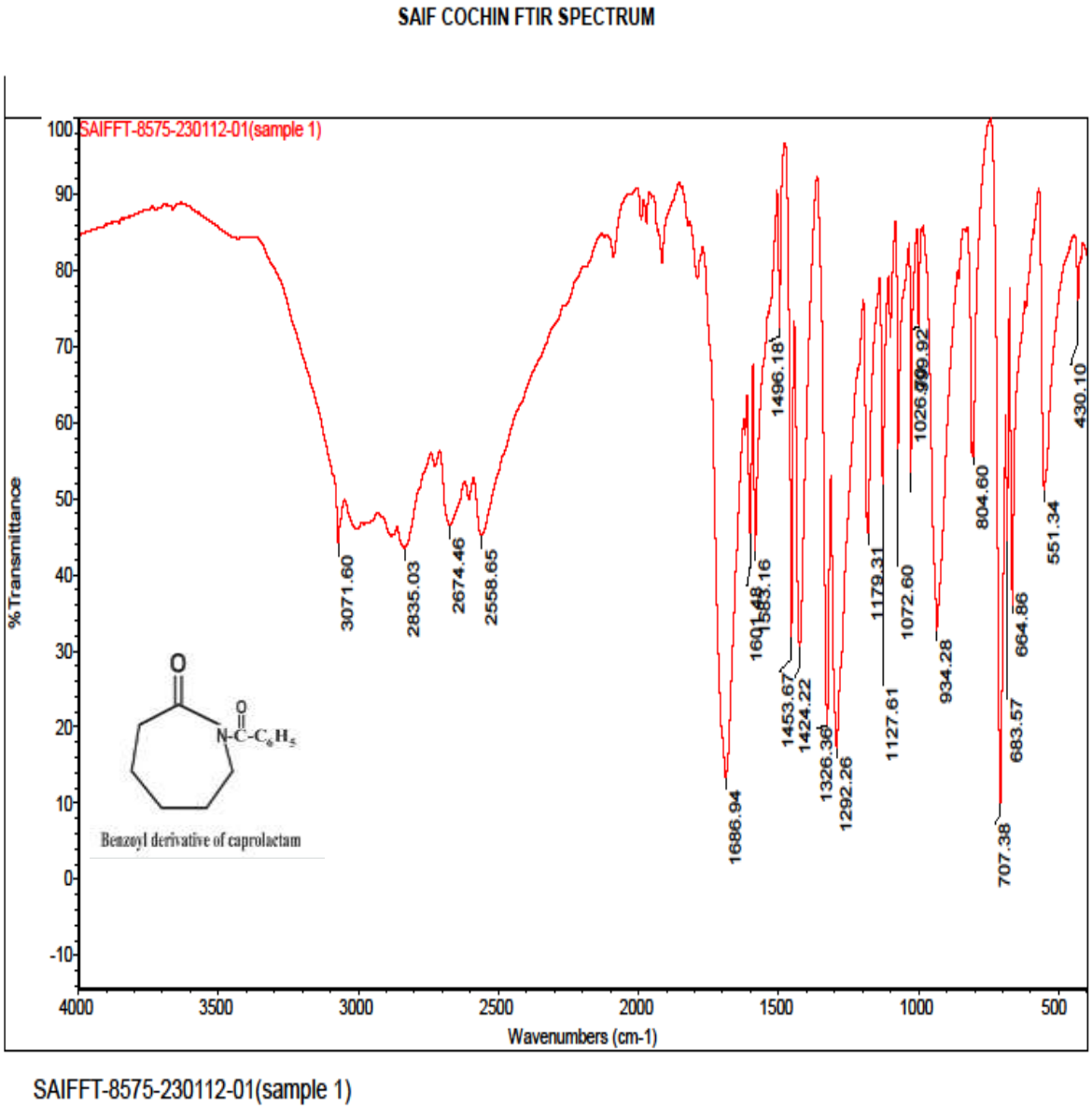

Figure 6: FT-IR of benzoyl derivative of caprolactam

Conclusion

It was observed that the maximum yield of monomer was obtained at 120 ^{0}C. Further by increasing the temperature, decrease in monomer yield was observed. Hydrolysis in the presence of IL [emim]BF_4 as a catalyst showed the maximum breakdown of nylon-6 in a shorter time than ILs [bmim]BF_4 and [bmim]PF_6. The hydrophilic nature of IL [emim]BF_4 and [bmim]BF_4 has shown an efficient tendency to catalyze the hydrolysis of nylon-6, while the hydrophobic nature of [bmim]PF_6 has the least tendency to catalyze the hydrolysis of nylon-6. These ILs play a crucial role in enabling water to interact with the polymer chains of nylon-6, entering the amide (-CO-NH-) linkage and breaking down into aminocaproic acid, which finally converts into caprolactam. Furthermore, among the [emim]BF_4 and [bmim]BF_4 hydrophilic ILs, it was observed that due to the smaller alkyl (non-polar) group, less viscosity and high

mobility of $[emim]^+$ and BF_4^- ions, its interaction with amide (-CO-NH-) linkages is more than $[bmim]BF_4$ which having larger alkyl group and more viscosity and less mobility of $[bmim]^+$ ion. Hence, $[emim]BF_4$ has more degradation efficiency than $[bmim]BF_4$. Due to the larger alkyl group and less diffusion tendency of both $[bmim]^+$, PF_6^- ions, and also water insoluble property of IL $[bmim]PF_6$ showed the least capacity to degrade waste nylon-6 and nyon-66. It was observed that as the alkyl group increases, the degradation capacity of ILs decreases.

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Conflict Of interest

The author declares that there is no conflict of interest.

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