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Optimisation Study of Efficiency of Ionic Liquids as Catalyst for Hydrolysis of Waste Nylon-66

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Abstract

With the increasing rate of production and usage of nylon-66, 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 liquids have shown efficient potential as catalysts for hydrolysis waste nylon-66 into dibenzoyl derivatives of hexamethylenediamine (DBHMD) and adipic acid. Ionic liquids [emim]BF_4, [bmim]BF_4, and [bmim]PF_6 successfully catalysed the hydrolysis of waste nylon-66 at different extents. 1.9 g (.1 mol) of [emim]BF_4 has shown the maximum catalytic efficiency during hydrolysis of nylon-66 at 120 °C when the reaction was performed for five hours. In comparison, 2.3 g (.1 mol) of [bmim]BF_4 and 2.8 g (.1 mol) of [bmim]PF_6 showed the maximum catalytic properties at 120 °C when the hydrolysis reaction was performed at seven and nine hours, respectively. However, the hydrophilic nature of IL [emim]BF_4 has shown more efficiency as a catalyst in the hydrolysis of waste nylon-66.

Key-words: catalyst, DBHMD, hydrolysis, ionic liquids (ILs), Nylon-66

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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 significantly improve human welfare through various valuable applications. However, due to their relative inertness and lack of biodegradability, plastics pose a significant treatment and disposal challenge in managing 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 comprise 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 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, There are several methods for disposing of waste nylon, but the most effective way to address this issue is to break it down into usable monomers. 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). The growing focus on adopting cleaner technologies has garnered significant attention from researchers and the industry.

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 favourable 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 considers them valuable solvents due to their exceptional solvent properties, stability at high temperatures, and low volatility. The melting point of ionic liquids (ILs) is lower than 100 °C or 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.

According to Kamimura A. et al. (2007), the counter anions in ionic liquids play a crucial role in the depolymerisation of polyamides. The study found that using DMAP (4-dimethylaminopyridine) as a catalyst and N-methylpyrrolidone as a solvent can achieve an impressive 86% yield of caprolactam after processing for six hours at 300 °C [32]. An ionic liquid [Bmim] [$FeCl_4$] can catalyse the depolymerisation of lignin to methyl p-hydroxycinnamate. Overall catalyst $[FeCl_4]^-$ 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 depolymerisation of the model compound lignin [33]. Ionic liquid [emim]BF_4 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]BF_4), 1-butyl-3-methylimidazolium tetrafluoroborate ([bmim]BF_4), and 1-butyl-3-methylimidazolium hexafluorophosphate ([bmim]PF_6). The study will compare the efficiency of these ionic liquids and optimise 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 various ionic liquids (ILs) used in this study were prepared in the laboratory. The 1-methylimidazole (99% pure) needed for synthesising the ILs was obtained from Sigma-Aldrich. All other chemicals used were of analytical grade. Solutions of 5N HCl and 5N NaOH were prepared using double-distilled water. **Experimental method**

To purify it, the nylon was treated with a 1 g/L solution of nonionic detergent at a temperature of approximately 70–80 °C for 6 to 7 hours. Following this treatment, it was thoroughly rinsed with distilled water to remove any surface impurities and then allowed to dry at a temperature of around 60–70 °C for 3 to 4 hours.

Next, 0.2 g of nylon-66 and 20 mL of 5N HCl were refluxed with 1.9 g (0.1 mol) of [emim]BF_4 as a catalyst for 5 hours at a temperature ranging from 80 to 120 °C. After the reaction cooled, the unreacted nylon-66 was separated, and the reaction mixture was neutralised with 5N NaOH to achieve a slightly alkaline pH (7–7.5). The addition of benzoyl chloride to this alkaline mixture precipitated a dibenzoyl derivative of hexamethylenediamine (DBHMD), which was then recrystallised using ethanol. After filtration, the filtrate primarily contained adipic acid, extracted with 20 mL of ethyl acetate in ten separate extractions. The ethyl acetate was then concentrated under reduced pressure using a rotary evaporator, yielding a colourless solid of adipic acid that was further recrystallised with methanol. The ionic liquid was also recovered using a rotary evaporator.

This experiment was repeated at different times and temperatures for an optimisation study. For comparison, 2.3 g (0.1 mol) of [bmim] BF_4 and 2.8 g (0.1 mol) of [bmim]PF_6 were used as catalysts for the hydrolysis of nylon-66. The reaction scheme for the hydrolysis of nylon-66 in the presence of ionic liquids as catalysts is illustrated in Figure 1.

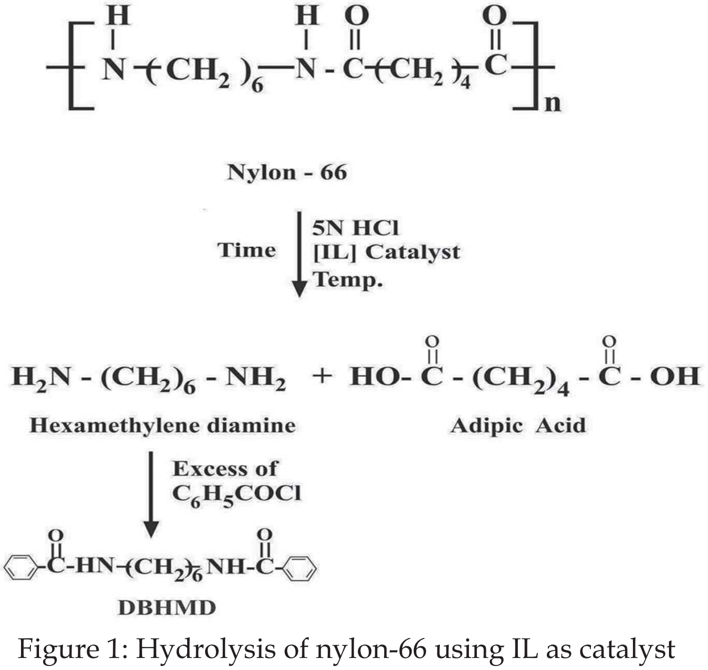


Figure 1: Hydrolysis of nylon-66 using IL as catalyst

Result and Discussion

Hydrolysis of nylon-66 using IL [emim]BF_4 as a catalyst

When nylon-66 was hydrolysed without a catalyst at 60°C for five hours, the yield of DBHMD and adipic acid was found to be 1.16 g and 0.006 g, respectively. At 120°C, the yields increased to 1.74 g of DBHMD and 0.013 g of adipic acid. However, it was the introduction of an ionic liquid (IL) catalyst that truly made a difference, significantly enhancing the yields of the monomers. Specifically, when nylon-66 was hydrolysed using 0.1 mol of [emim]BF_4 at 120°C for five hours, the maximum yields were recorded: 3.74 g of DBHMD and 0.098 g of adipic acid. At 140°C, there was a slight decrease in the yields. Additionally, it was noted that hydrolysis of nylon-66 at 100°C for five hours produced fewer monomers compared to the reaction at 120°C for four hours, both conducted with the same catalyst concentration. This indicates that as the temperature increases, the degradation time decreases. The results are summarised as follows in **Table 1**.

.2g of nylon-66 + 1.9g (0.1 mol.) of [emim]BF_4					
Sr. no.	**Conc. of [emim]BF_4 (g)**	**Time (hrs.)**	**Temp (^0C)**	**DBHMD (g)**	**Adipic acid (g)**
Different temp.					
1	0	5	60	1.13	.006
2	0	5	120	1.74	.013
3	1.9	5	80	2.62	.051
4	1.9	5	100	3.11	.063
5	1.9	5	120	3.74	.098
6	1.9	5	140	3.69	.091
Different time					
7	1.9	4	120	3.41	.072
8	1.9	3	120	2.83	.039

Table 1: Hydrolysis of nylon-66 in [emim]BF4 catalyst

Hydrolysis of nylon-66 using IL [bmim]BF_4 as catalyst

The hydrolysis of nylon-66 in 0.1 mol of [bmim]BF_4 catalyst is summarised in Table 2. It was observed that the ionic liquid [bmim]

BF_4 catalysed the hydrolysis of waste nylon-66 to a lesser extent than [emim]BF_4. When we performed the hydrolysis of nylon-66 for five hours at 120 °C using 0.1 mol of [bmim]BF_4 as a catalyst, we obtained a smaller amount of DBHMD and adipic acid compared to using 0.1 mol of [emim]BF4 under the same reaction conditions. Additionally, even after extending the reaction time to seven hours with 0.1 mol of [bmim]BF_4, the yield of monomers remained lower at 120 °C. This indicates that [bmim]BF_4 is less efficient as a catalyst for the hydrolysis of nylon-66 than [emim]BF_4.

.2g of nylon-66 + 2.3g (0.1 mol.) of [bmim]BF_4					
Sr. no.	**Conc. of [bmim] BF_4 (g)**	**Time (hrs.)**	**Temp (^{0}C)**	**DBHMD (g)**	**Adipic acid (g)**
Different temp.					
1	0	7	60	1.19	.008
2	0	7	120	1.93	.013
3	2.3	7	80	1.98	.028
4	2.3	7	100	2.68	.033
5	2.3	7	120	2.99	.048
6	2.3	7	140	2.87	.032
Different time					
7	2.3	6	120	2.71	.041
8	2.3	5	120	2.23	.026

Table 2: Hydrolysis of nylon-66 using [bmim]BF_4 as catalyst

Hydrolysis of nylon-66 using IL [bmim]PF_6 as catalyst

As shown in Table 3, nylon-66 was hydrolysed at varying temperatures and durations while 2.8 g (0.1 mol) of [bmim]PF6 was used as a catalyst.

.2g of nylon-66 + 2.8g (0.1 mol.) of [bmim]PF_6					
Sr. no.	**Conc. of [bmim]PF_6 (g)**	**Time (hrs.)**	**Temp (^{0}C)**	**DBHMD (g)**	**Adipic acid (g)**
Different temp.					
1	0	9	60	1.20	.012
2	0	9	120	2.06	.011
3	2.8	9	80	1.32	.019

4	2.8	9	100	2.01	.020
5	2.8	9	120	2.31	.024
6	2.8	9	140	2.26	.019
Different time					
7	2.8	8	120	2.16	.021
8	2.8	7	120	1.55	.016

Table 3: Hydrolysis of nylon-66 using [bmim]PF_6 as catalyst

When we compare the hydrolysis of nylon-66 in the absence of IL and with IL as a catalyst at 120 °C for nine hours, we observe a significant but slight difference in DBHMD and adipic acid yield. In the absence of IL, the yield of DBHMD and adipic acid was 2.26 g and .011 g, respectively, while in IL as a catalyst, the yield was 2.31g and .024g, respectively. These results underscore the modest efficiency of [bmim]PF_6 as a catalyst in the hydrolysis of nylon-66, providing valuable insights for further research. Our experimental work revealed that all three ILs effectively catalysed the hydrolysis of nylon-66 to varying degrees. We observed that the maximum yield of monomers was achieved at 120 °C, and as the temperature was further increased to 140 °C, the yield of monomers decreased. This temperature sensitivity is a critical factor in the hydrolysis process. The higher mobility of $[emim]^+$ and BF_4^- ions, due to their lower viscosity, facilitated a quicker hydrolysis of nylon-66. Conversely, the higher viscosity and lower mobility of $[bmim]^+$ and PF_6^- ions resulted in a longer hydrolysis time. Among the three ILs, due to the hydrophilic nature and less viscosity of [emim]BF_4, it enhances the water molecule to interact with the amide (-CO-NH-) linkages of nylon-66 and accelerate the hydrolysis of nylon-66 and showed more efficiency as the catalyst. Due to the hydrophobic nature and high viscosity of [bmim]PF_6, it does not initiate the water molecules to penetrate the nylon-66 matrix, resulting in less degradation of nylon-66 and less efficiency as the catalyst. IL [bmim]BF_4 has moderate solubility in water and moderate viscosity. Hence, it has shown moderate catalytic properties. ILs are not directly involved in breaking amide linkages, but they help the water molecules enter the nylon matrix, resulting in the cleavage of amide linkage. The following charts illustrate the variation in DBHMD and adipic acid amounts across different ionic liquids.

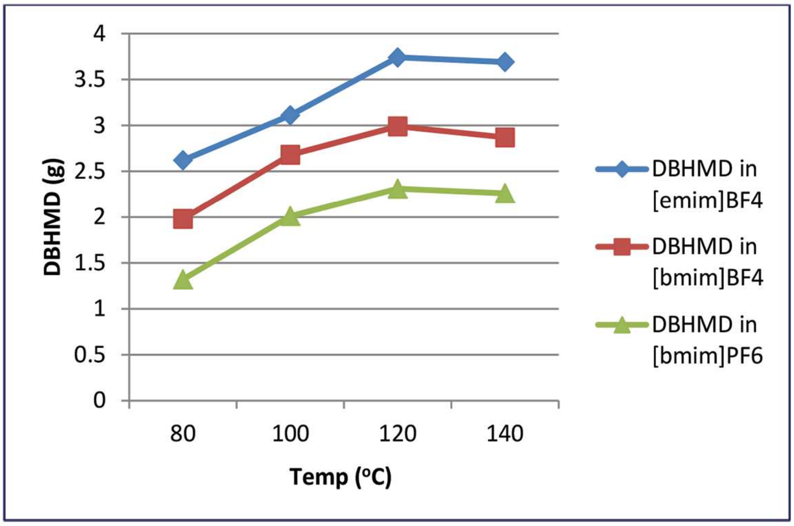


Figure 1: Variation of DBHMD in different ILs with the temperature

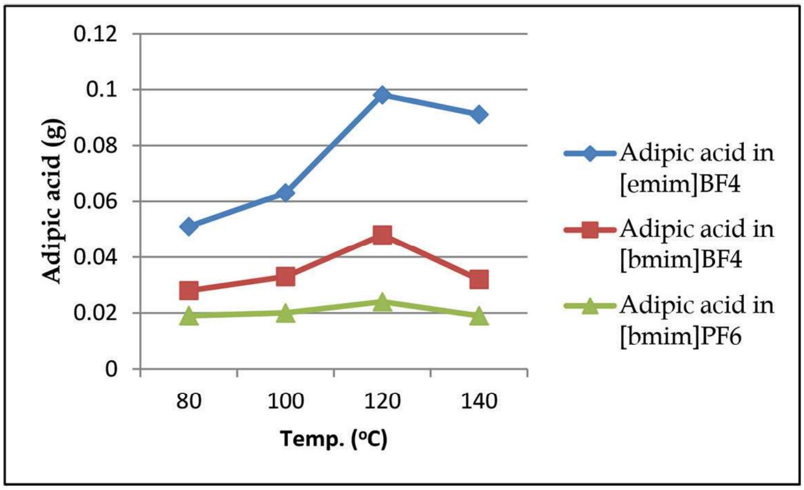


Figure 2: Variation of adipic acid in different ILs with the temperature

Hydrolysis of nylon-66 follows through the following mechanism.

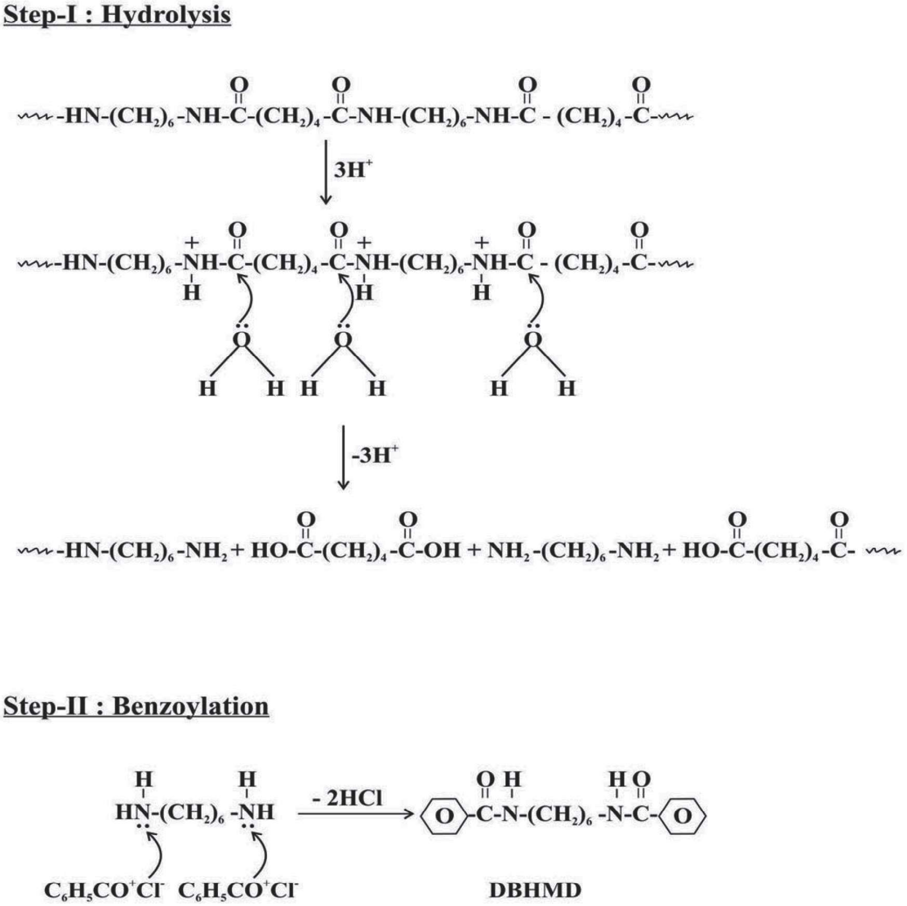


Figure 3: Mechanism of hydrolysis of nylon-66

Characterisation of Monomers

Both monomers DBHMD and adipic acid were characterised by FT-IR and 1H-NMR techniques. Both the monomers were washed with water and recrystallised with ethanol. The melting points of DBHMD and adipic acid were recorded as 58°C and 150°C, respectively.

FT-IR of DBHMD

FT-IR cm^{-1}: 3305.37 (N-H stretching), 1698 (C=O stretching), 1627, 1530, 1479, 1374 (C=C stretching in benzoyl group), 1416 (C-C stretching in CH_2 groups), The two peaks at 788 and 861 corresponds to mono-substituted benzene ring. Due to the symmetrical structure of BDHMD, only one peak for N-H and C=O stretching was obtained.

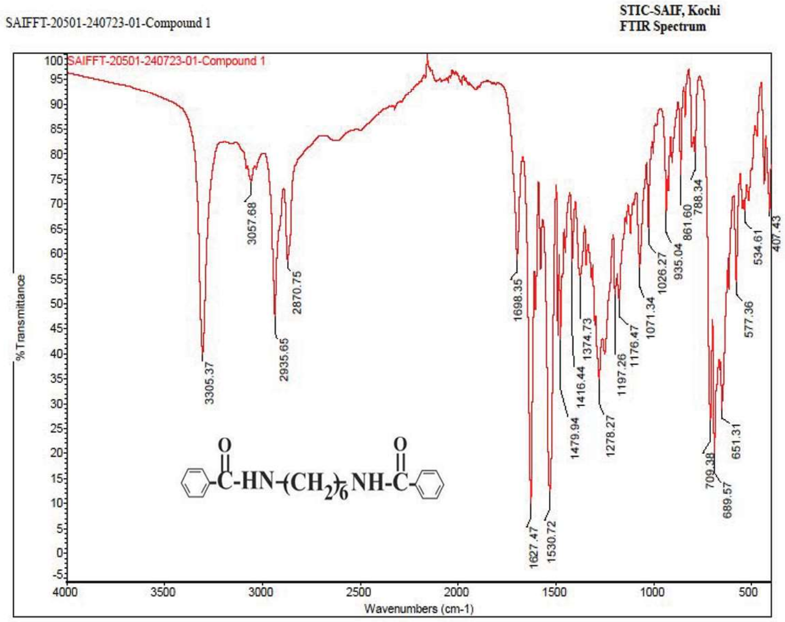
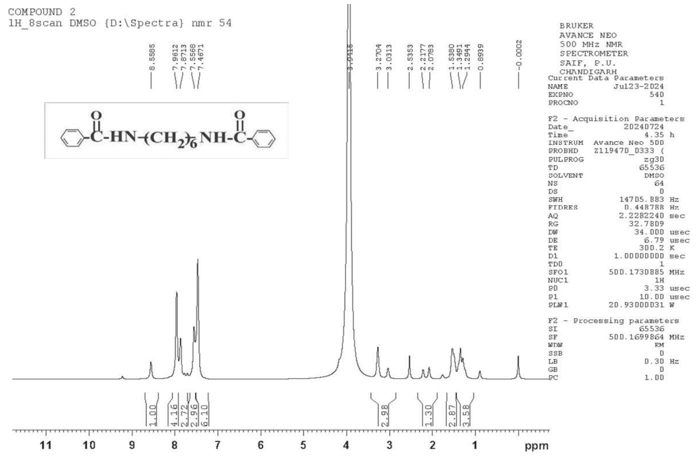


Figure 4: FT-IR of DBHMD

^{1}H-NMR of DBHMD

DBHMD has a symmetrical structure featuring two benzoyl groups and six methylene (>CH2) groups. Two distinct multiplets correspond to the protons of the aromatic ring and the twelve methylene protons.

^{1}H-NMR (δ ppm) (500 MHz, DMSO-d$_6$): 7.46-8.55 (m, 5H, Ar-H), 1.29-3.94 (m, 12H, 6 >CH$_2$)



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500 MHz NMR
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SAIF, P.U.
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PROCNO                 1

F2 - Acquisition Parameters
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PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    64
DS                     0
SWH            14705.883 Hz
FIDRES          0.448788 Hz
AQ             2.2282240 sec
RG               32.7809
DW                34.000 usec
DE                  6.79 usec
TE                 300.2 K
D1            1.00000000 sec
TD0                    1
SFO1         500.1730885 MHz
NUC1                  1H
P0                  3.33 usec
P1                 10.00 usec
PLW1         20.93000031 W

F2 - Processing parameters
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SF           500.1699864 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
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Figure 5: ^{1}H-NMR of DBHMD

FT-IR of adipic acid

FT-IR (cm^{-1}): Adipic acid exhibits a broad absorption band in the 2500-3000 cm-1 region, attributed to -OH stretching in the carboxylic group (-COOH).

2662 (-OH stretching in –COOH), 2887-2951 (C-H stretching in >CH$_2$), 1682 (>C=O stretching), The bands below 1500 cm^{-1} are due to C-O and C-C stretching and bending, respectively.

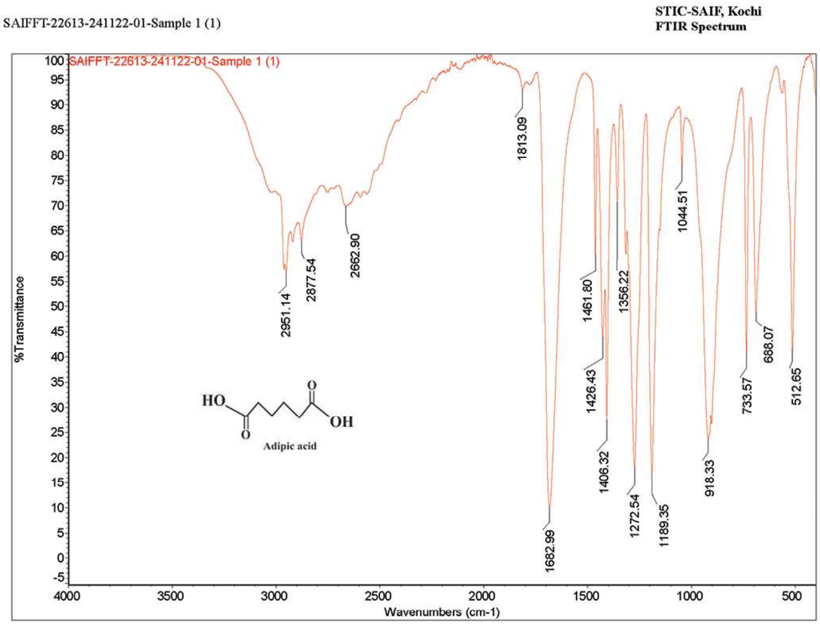

Figure 6: FT-IR of adipic acid

^{1}H-NMR of adipic acid

The carboxylic (-COOH) group in adipic acid was recognised by the singlet peak above 10 ppm. Adipic acid contains four methylene (>CH$_2$) groups. The two >CH$_2$ groups show a peak at a higher δ value than the middle two >CH$_2$ groups.

^{1}H-NMR (δ ppm) (500 MHz, DMSO-d$_6$): 1.53 [t, 4H, -CH$_2$-C**H**$_2$-C**H**$_2$-CH$_2$-], 2.20 [t, 4H, -C**H**$_2$-CH$_2$-CH$_2$-C**H**$_2$-], 11.97 [s, 1H, -COO**H**]. The DMSO-d$_6$ displayed a singlet peak at δ2.52 ppm.

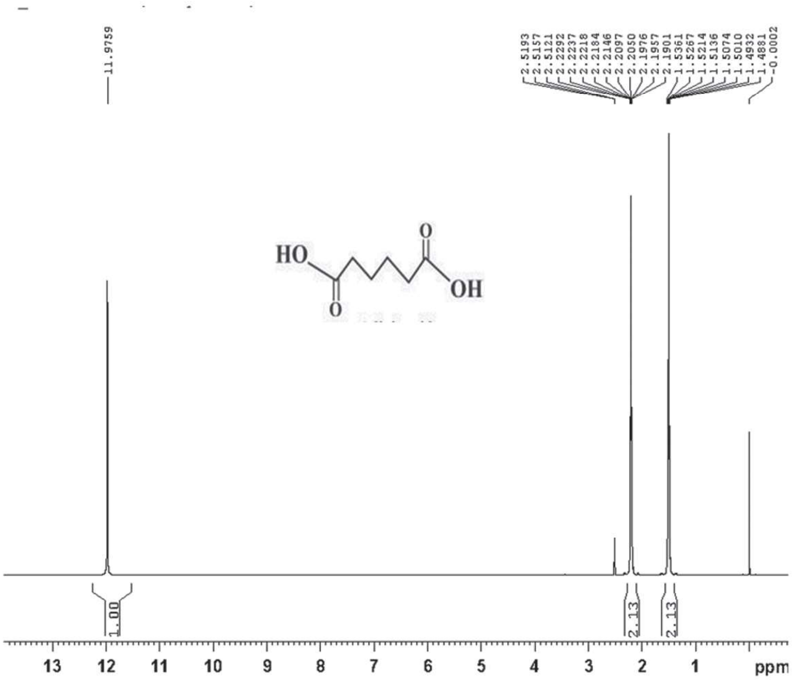

Figure 7: 1H-NMR of adipic acid

Conclusion

The hydrolysis of nylon-66 using [emim]BF$_{4,}$ [bmim]BF$_4$ and [bmim]PF$_6$ as catalysts provides valuable insight into a novel approach for the breakdown of polyamide and the disposal of this widely used synthetic polymer. All these three ionic liquids have shown the successful hydrolysis of nylon-66 into the dibenzoyl derivative of hexamethylenediamine (DBHMD) and adipic acid, and these monomers were confirmed by FT-IR and ^{1}H-NMR techniques. In this experimental work, IL [emim]BF$_4$ has shown maximum efficiency as a catalyst in the hydrolysis of nylon-66 and breaks down the amide linkages at a maximum extent due to the maximum solubility in water. Meanwhile, ILs [bmim]BF$_4$ and [bmim]PF$_6$ have shown less efficiency as catalysts due to moderate and least solubility in water, respectively. The hydrolysis of nylon-66 using ionic liquids (ILs) as a catalyst provides a viable solution to address the environmental pollution associated with the disposal of waste nylon-66. This material is durable and persistent in the environment, and this hydrolysis method does not produce any harmful by-products. Parameters such as temperature and reaction time play a significant role in optimising the degradation process. Due to the low volatility and reusability of ionic liquids, they do not create any additional burden on the environment. It was regenerated at the end of the reaction without changing its efficiency, and it can be used four to five times.

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Conflict Of Interest

The author declares that there is no conflict of interest.

Authors' Contribution

Both authors were equally involved in performing experimental work and preparing the manuscript.

Data Availability Statement

Data is generated by the study undertaken by the author.

Ethics Approval Statement

This research does not involve human participants, animal subjects, or any material that requires ethical approval.

Consent Statement

This study did not involve human participants, and therefore, informed consent was not required.

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