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ORIGINAL ARTICLE

Depolymerization of Nylon Waste by Hydrolysis using surfactant and its Kinetic Study

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Abstract

Objectives: Polymers are nonbiodegradable, and they have become a big environmental issue; hence, many recycling processes have been introduced. By recycling nylon 6,6 waste, the process can reduce the environmental impact which is associated with the production of new materials. Depolymerization offers opportunities for the development of innovative chemical recycling process. **Methods:** Nylon waste is depolymerized through acidic hydrolysis concentration of HCL 5N in presence of catalysts sodium lauryl sulphate at favourable temperature 80^0C shows the changes in the amount of regaining of the monomer that is hexamethylene diamine (HMD) into Dibenzoyl derivative of hexamethylene diamine (DBHMD) in 120 min. The product was characterised by FTIR analysis. **Findings:** Amount of DBHMD is directly proportional to the amount of HMD produced which can be used as the criteria to determine the efficiency of the method. The nature of catalyst and their amount both are found to affect the yield of product by significant value. About 84% product (DBHMD) have been obtained by taking 0.03 g of SLS and 3g of nylon waste was refluxed by heating about 120 min and its rate of reaction was found to be 8.90×10^{-3} min^{-1}. Kinetics shows pseudo 1^{st} order. **Novelty:** This research provides an easy and quick depolymerisation of such nonbiodegradable wastes Nylon-6,6 compared to other processes.

Keywords: Depolymerization; Nylon Waste kinetic; Sodium Lauryl Sulphate; Acid Hydrolysis

1 Introduction

Plastics have become very essential part of human life due to its excellent properties such as durability, chemical resistance, fire resistance, ductility, light in weight, flexibility and many more(?). There are many types of plastics such as polythene, polyurethane, polyamide, etc which are commonly used in our daily life. Despite of its excellent properties, it has worse effect also due to its nonbiodegradable nature, its accumulation has emerged as global ecological problems(?). Plastics takes lots of year to degrade hence an alternative method for clean environment is recycling of such plastics for this many methods have been developed such as pyrolysis, hydrolysis, aminolysis etc(?),(?),(?).

The recycling process can be done through various steps such as collecting plastic waste, sorting, cleaning, drying, cutting into small sizes, chemical processing, product, quality checking and many more and this require lot of equipment, labour, money and most important it requires time(?). There are many types of catalyst which were used in many works for getting better result such as sub-critical water, ethylene glycol solution, enzymes, etc(?),(?),(?).

Very few works have been performed by using surfactants as catalyst for depolymerization of plastics such as nylon-6,6. In this work we have depolymerized nylon waste by acid hydrolysis, molecular viscosity of nylon-6,6 [nylon waste], IR study and kinetics of dibenzyl derivative of hexamethylene diamine using surfactant has been studied.

2 Methodology

2.1 Material and apparatus

Nylon waste was collected from waste materials, crushed into small particles, cleaned in detergent and then with abundant water and then dried in an oven and then used for further process. 0.5 N HCl was used for hydrolysis and after process solution was neutralised by using 0.5 N NaOH. HCl and NaOH pallets and sodium lauryl sulphate (used as catalyst) were of commercial grades. Benzoyl chloride of commercial grade was used for making derivative of the monomer. Round bottom flask, water condenser etc were used.

2.2 Molecular viscosity of nylon waste (polyamide 66)

Various concentration of Nylon waste was dissolved on m-cresol. M- cresol used as solvent. Through viscometer time flow of various concentration of nylon waste was measured using m - cresol (solvent) and plotted a graph between "concentration and $[\eta sp]/C$" on X and Y axis respectively, using formula "$[\eta sp]/C = KM^{\alpha}$", here value of solvent for K and a is 2.4018×10^{-3} and 0.6121 respectively, intercept along with average molecular weight (AMW) was determined.

2.3 Depolymerizing Nylon-6,6 waste using SLS

Sodium lauryl sulphate is utilized as catalyst in this context. Due to its anionic character it helps to depolymerize nylon-6,6 waste in acidic medium. By using drop count method CMC of sodium lauryl sulphate does not have any relation with the reaction because depolymerization takes place at higher level than that of CMC of SLS.

Optical parameters were determined gravimetrically for converting nylon-6,6 into its monomer. 3g of nylon-6,6 waste were acid hydrolysed using 0.5N HCl using various amount of catalyst i.e. SLS (0.1g- 1g) for various time interval (30 min-240 min) at 80^0 C. Reaction mixture was then filtered and neutralised by using 0.5N NaOH solution and tested by using red litmus paper which gets turned into blue which indicates the neutralisation of the reaction mixture. Benzoyl chloride was then added till white precipitate is not formed and odour gets diamine (DBHMD). DBHMD was then recrystallised by ethanol. It was found that by taking 0.03 g of SLS, 3g of Nylon-6,6 get depolymerized and gives highest yield of DBHMD by refluxing using water condenser for 120 min at 80^oC.

3 Result and Discussion

3.1 Molecular viscosity of Nylon-6,6 Waste

Through the viscosity method, the AMW of nylon waste was assessed by creating a graph with "concentration and $[\eta sp]/C$" on the X and Y-axis respectively featuring an intercept 1.2250. Utilizing the formula "$[\eta sp]/C = KM^{\alpha}$," where K is valued at 2.41×10^{-3} and α equals 0.61. AMW of nylon waste = 27480.

3.2 Depolymerizing Nylon waste using SLS

Various amounts of SLS (0.01g-0.07g) was refluxed with 3g of Nylon-6,6 waste in acidic solvent of 0.5N HCl at constant temperature 80^0C throughout the reaction process for various time intervals (30 min-240 min) and then neutralised using 0.5 N NaOH and the filtered and few amount of benzoyl chloride was added till odour disappears and found that by taking 0.03g of SLS and for 120 min heating , the reaction mixture gets converted into its product i.e. DBHMD with highest yield. Figure 1 shows first increase and then decrease in the yield of DBHMD. Highest yield obtained by taking 0.03g SLS and gives 84.3% yield. Figure 2 shows the maximum time required is 120 min for converting nylon waste into its monomer.

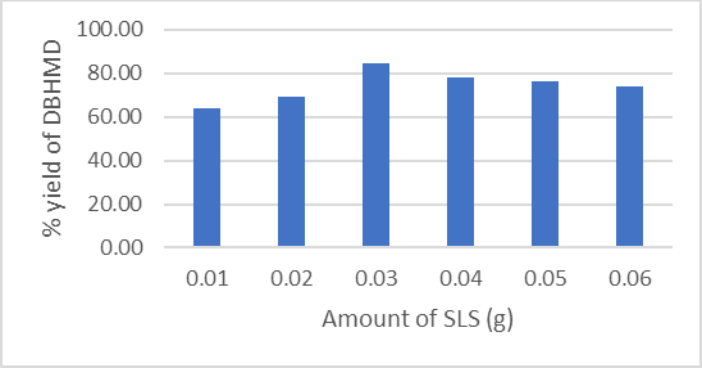

Fig 1. % yield of DBHMD obtained by taking various amount of SLS

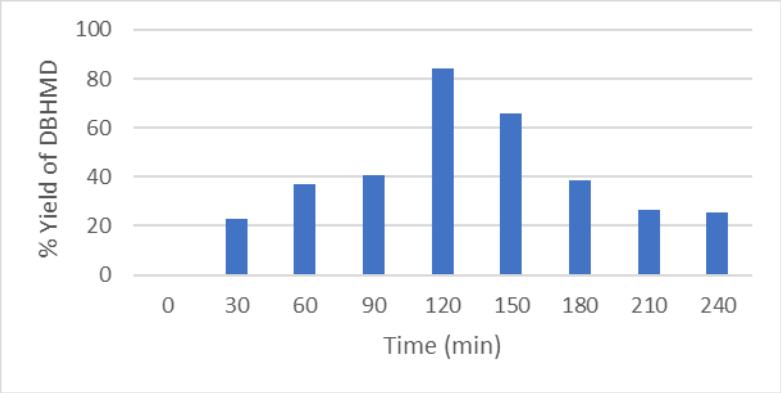

Fig 2. Various time intervals required for converting nylon waste into its monomer

3.3 Kinetic analysis of the Nylon waste Depolymerization

In order to turn nylon waste into its product, DBHMD, we used nylon-6,6 waste, HCl, H_2O, and SLS in this investigation. The amount of product obtained, as indicated in table 1, was used to calculate the amount of nylon waste reacted and the amount of nylon waste unreacted. From kinetics equation it is noted that depolymerization of nylon-66 (nylon waste) shows pseudo first order reaction which is shown by following equation,

$$\frac{-d[Nylonwaste]}{dt} = k[Nylon][HCl]^m[H2O]^n[SLS]^p \tag{1}$$

Since very small amount of SLS was used for reaction and more amount of H2O and HCl was used therefore their concentrations were taken negligible and hence above equation written as,

$$\frac{-d[Nylonwaste]}{dt} = k[Nylon-6,6waste] \tag{2}$$

Let [Nylon waste]$_o$ be the starting amount of Nylon-6,6 and [nylon waste]i be the amount at a certain point in time. Integral (**eq. 2**) is displayed below as,

$$\ln[Nylonwaste]_o[Nylonwaste]_i = k't \tag{3}$$

Since nylon-6,6 waste is solid, it may be measured in weight. The third equation above can now be expressed as follows:

$$\ln\frac{a}{a-x} = k't \tag{4}$$

where (a-x) is the "weight at a certain time interval" and an is the beginning weight [?]. We can state that the first-order reaction's rate **equation (11)** is,

"$K = (2.303/t)\log(a/(a-x))$;"
Above equation can be rearranged as

$$\log(a-x) = \frac{-kt}{2.303} + \log a \tag{5}$$

The eq. can be rewritten, "$\log(a/(a-x)) = Kt/2.303$"; which can be represented as $y = mx$. From table 2 and Figure 3 show a straight line passing through origin which indicates that reaction shows first order kinetics and reaction constant obtained 8.90×10^{-3} min^{-1}.

Table 1. Amount of DBHMD obtained at various time interval

Sr no.	Time (min)	Weight of nylon waste (a)	Weight of SLS	Weight of DBHMD	Weight of reacted nylon(x)	Weight of unreacted nylon waste [A] or (a-x)	% Yield
1	0	3	0.03	0	0	3	0
2	30	3	0.03	0.99	0.68	2.31	22.99
3	60	3	0.03	1.60	1.10	1.89	36.99
4	90	3	0.03	1.75	1.21	1.78	40.63
5	**120**	**3**	**0.03**	**3.62**	**2.53**	**0.46**	**84.34**
6	150	3	0.03	2.83	1.97	1.02	65.76
7	180	3	0.03	1.66	1.15	1.84	38.61

Table 2. Kinetis order of depolymerization of Nylon-6,6

Time (min)	log(a-x)	a/a-x	log(a/a-x)
0	0.47	1.00	0.00
30	0.36	1.29	0.11
60	0.27	1.58	0.20
90	0.25	1.68	0.22
120	**-0.32**	**6.38**	**0.80**
150	0.01	2.92	0.46
180	0.26	1.62	0.21

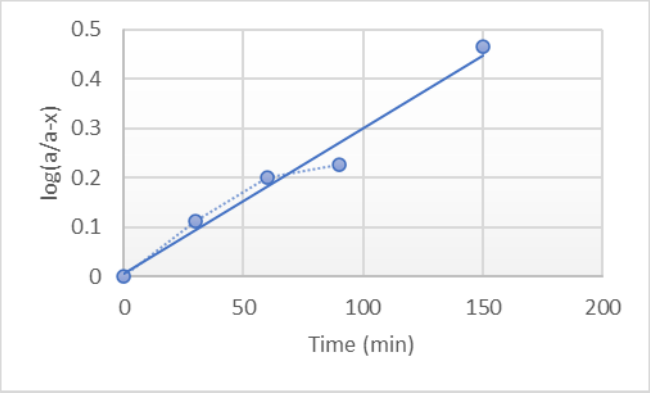

Fig 3. Graph plotted between "Time (X- axis) and log (a/(a-x)) (Y-axis)"

3.4 Analysis of DBHMD using FTIR Spectroscopy

FTIR spectra assisted in determining that the FT-IR spectra value of controlled product "DBHMD" and the acid hydrolysis-derived product "DBHMD" from nylon-6,6 waste have similar peaks, as shown in figures 4 (?) and Figure 5. Every band displayed

is clear, indicating the presence of H-bonding. Various bands detected at 2854 cm^{-1} show the existence of C-H stretching, 3304 cm^{-1} show the existence of N-H stretching, 1417 cm^{-1} indicates existence of C-C stretching, 1687 cm^{-1} show the existence of Carbon double bond Oxygen stretching. The intense peak on the left side of the 3304 cm^{-1} value show N-H stretching is amide functional group, combining characteristics of amine and ketone with N-H and C=O bonds[?]. Numerous peaks ranging from 1500-400 cm^{-1} indicate various vibration types in molecules, including unsimilar stretching patterns. The FT-IR spectra for derived product "DBHMD" as well as controlled DBHMD are similar, therefore the same explanation applies.

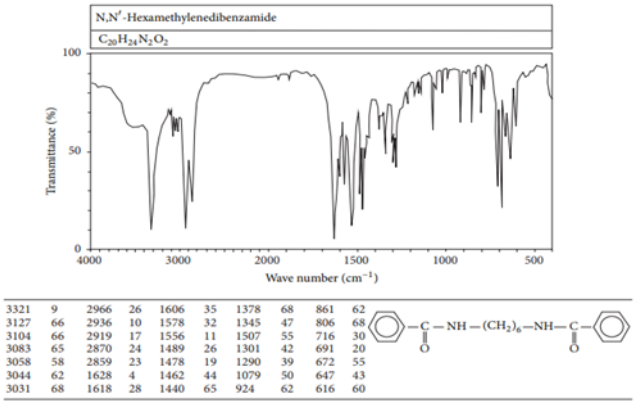

Fig 4. FT-IR Spectral data of DBHMD (controlled)

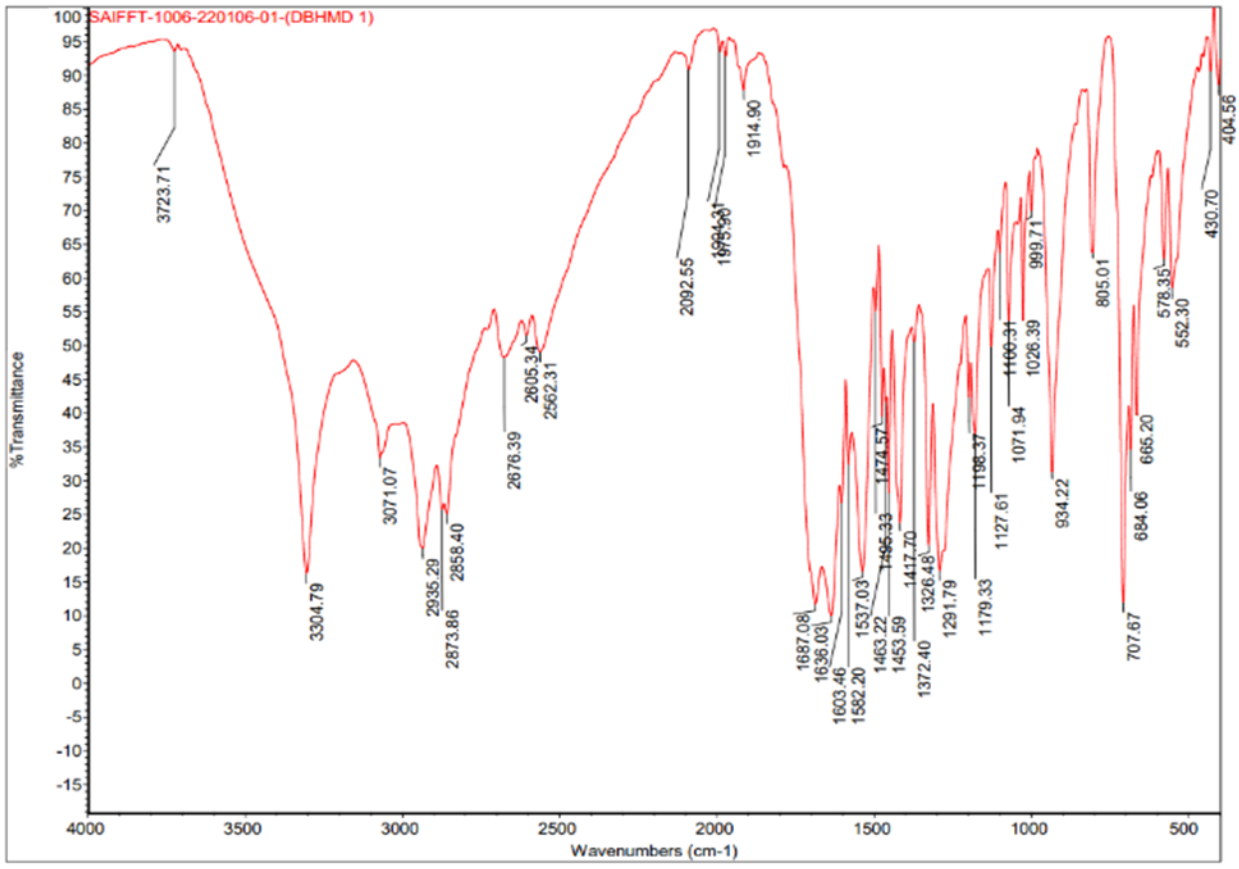

Fig 5. FT-IR Spectra of product DBHMD

4 Conclusion

With the use of sodium lauryl sulfate surfactant, the study displays the reaction rates of nylon-6,6 hydrolysis. Using the viscosity approach, we first calculated the molecular weight of the discarded nylon-6,6 waste. The drop count method was used to measure the critical micelle concentration (CMC) of SLS, an anionic surfactant that acts as a catalyst. The study found no relationship between the CMC of SLS used in the depolymerization of nylon waste. The optimal amount of SLS catalyst for the highest product DBHMD yield is 0.03g, which was obtained by heating different amounts of SLS (0.01g to 0.07g) at 80 ^{0}C for 120 minutes. When nylon-6,6 waste was depolymerized using 0.03g of catalyst over different time intervals at 80°C, it was discovered that the amount of product produced after 120 minutes of heating was significantly higher than at other time intervals. Because of the surplus water, a straight line that passes through the origin exhibits first-order kinetics and a pseudo-first-order reaction. The reaction rate constant is 8.90×10^{-3} min^{-1}. The melting point of the product DBHMD is 157 °C. The product DBHMD FTIR spectrum is twinning to the standard DBHMD.Nylon-6,6 waste degradation reduces the environmental pollution hence obeys principles of green chemistry. Future study should be on interaction of different anionic surfactants with nylon-6,6 waste at different parameters.

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