STUDIES ON THE PRODUCTION OF P-PHENYLENEDIAMINE FROM PET WASTE BASED ON AN EFFICIENT METHOD AND INCREASING THE REACTION YIELD

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Abstract. *This study focuses on the synthesis of p-phenylenediamine using polyethylene terephthalate (PET) waste as a raw material. Initially, terephthalic acid was recovered from PET waste via an alkaline hydrolysis method. The obtained terephthalic acid was then amidated with urea under high pressure in the presence of a catalyst. Subsequent chlorination was carried out under controlled neutral conditions. In the following step, an alkali treatment based on the Hofmann rearrangement was applied, and the resulting product was filtered and purified from salts through extraction with organic solvents.*

Throughout the process, key parameters such as catalyst type, temperature, pH, chlorine flow rate, and pressure were systematically varied to maximize the reaction yield. The composition and properties of the synthesized p-phenylenediamine were characterized using IR spectroscopy, SEM, TG, and DTA analyses. Optimal conditions were established for the amidation step, with a mass ratio of terephthalic acid to urea of 1:1.4. The reaction temperature was 185 ºC, the pressure was 6 atm, 1.6% (K1) catalyst was used, and the reaction took 3 hours. For the chlorination process, the reaction took 3.5 hours, 5ºC, pH 7, and a chlorine flow rate of 4 g/min were considered the most optimal options, and the reaction yield of p-phenylenediamine was 95%.

Keywords: *P-phenylenediamine, PET (polyethylene terephthalate), amination, chlorination, catalyst, reactor autoclave, Hofmann reaction*

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1. Introduction

The global production and accumulation of polyethylene terephthalate (PET) waste continue to rise each year. Although numerous recycling methods have been developed, the demand for effective PET recycling is growing rather than declining. PET is a non-toxic, lightweight, transparent, and odorless polymer characterized by strong chemical stability [1]. It is extensively used in food storage and packaging, engineering plastics, textiles, and light industry applications [2].

In 2021, global PET production reached 80.9 million tons, and it is projected to increase to 114.7 million tons by 2028 [3]. Due to its inherent chemical stability, PET requires centuries to fully decompose under natural conditions [4]. Moreover, its degradation produces microplastics and other harmful fragments that threaten the health and safety of both aquatic and terrestrial organisms [5]. Consequently, efficient PET recycling has become an urgent priority to ensure sustainable resource utilization and to mitigate environmental hazards [6].

Chemical processing is used to produce important chemicals from PET. Depolymerization of PET is a promising method for this [7]. There are various methods for the production of terephthalic acid (TP) and many other chemicals. For example, hydrolysis, acid and alkali treatment, as well as methanolysis and glycolysis, have been developed [8]. Ammonolysis is also used to produce the valuable terephthalamide (TPA), which is a key raw material for the synthesis of pesticides, organic substances, and additives in composite materials [9].

The conventional degradation of PET by ammonia solution at room temperature is not favored due to its low reaction efficiency and long reaction time [10]. Even when zinc acetate is used as a catalyst, PET recycling requires 30 days [11]. By depolymerizing PET by ammonolysis in ethylene glycol (EG) at high pressure (0.1–1.0 MPa) of ammonia solution, it is possible to produce 50% to 95% TPA [12]. However, the addition of a large amount of EG complicates the separation and purification processes, thereby increasing the overall process costs [13]. Considering these shortcomings, we focused on obtaining a higher-quality product and developing an efficient method [14].

Also, much work has been done to synthesize p-phenylenediamine, which is used as a raw material in the production of various pigments, antioxidants, polymers, and many other products [15]. In fact, there are several different methods for obtaining p-phenylenediamine [16]. For example, it is synthesized by hydrogenation of dinitrobenzene and by using aniline as a raw material in industry [17]. In addition, there is the reaction of terephthaloyl chloride with metal azides [18]. The only drawback of this method is that it increases the cost of the product due to the chlorinating and aminating agents used [19].

Another popular method for synthesizing p-phenylenediamineis is the reaction of terephthalamide with sodium hypochlorite, i.e., the Hofmann reaction [20]. This method is very convenient because the materials used are relatively cheap. Therefore, it does not increase the cost of the product, but the only drawback of this method is the reaction yield [21, 22]. High product yields cannot be achieved in this reaction. Considering these disadvantages, we have conducted research on obtaining p-phenylenediamine through a method that achieves high reaction yields and is economically efficient [23, 24].

2. Experimental part

2.1 Reagents and instruments. For the study, cleaned and crushed pieces of waste PET bottles and articles, urea, sodium hydroxide solution, pure chlorine gas, and organic solvents toluene, acetone, and benzene were used. A 5 L high-pressure HNZXIB reactor autoclave made of ordinary stainless steel and a SHIMADZU IR spectrophotometer for determining the chemical bonds of the resulting product and valence and deformation changes. A MIRA 2 LMU scanning electron microscope and a TGA-50/TG series thermogravimetric analyser for obtaining electron microscopic images, porosity, and elemental analysis.

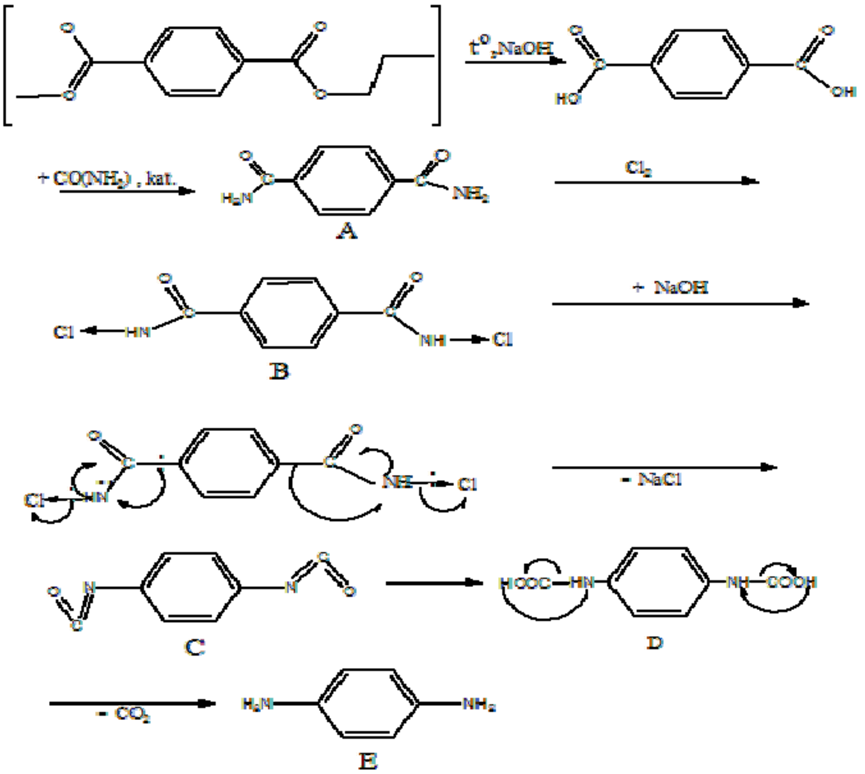


Fig. 1. Reaction mechanism and pathway

2.2 Reaction Mechanisms. The reaction mechanism and route for obtaining p-phenylenediamine from PET waste are shown in Fig. 1.

Using an alkaline method of chemical processing, terephthalic acid was obtained by breaking down long polymer chains of polyethylene terephthalate. The -OH part of the carboxyl group in the obtained terephthalic acid was converted to an amide by amination. The obtained terephthalamide was chlorinated in a neutral medium, and in this process, chlorine was replaced by one hydrogen of the amide, forming substance B. Substance B undergoes a Hoffman rearrangement reaction in an alkaline medium, as a result of which deprotonation occurs, displacing the hydrogen in -CO-NHCl, and the formation of a negative charge on the nitrogen causes the formation of the chloramide anion $-CO-N^{-}-Cl$. As a result of the release of chlorine from the formed anion, substance C is formed, i.e., isocyanide. Since the reaction takes place in an aqueous medium, substance C undergoes hydrolysis as a result of the action of water molecules on carbon and nitrogen, forming substance D. Since substance D is unstable, decarboxylation occurs, releasing carbonic anhydride, and the final product is substance E (p-phenylenediamine) [25].

2.3. Preparation of terephthalamide (TPA) in the presence of simple inorganic catalysts. To obtain terephthalamide, terephthalic acid, and urea were used, and sodium chloride or copper (II) sulfates were used as catalysts. The process is carried out in a 5-liter reactor autoclave at a temperature of 185°C under a pressure of 5-6 atm. 50 g of urea, 72 g of terephthalic acid and 2 g of sodium chloride are placed in the reactor autoclave. The reaction lasts for 3 hours. After 4 hours, i.e., when urea is completely decomposed into carbon dioxide and ammonia and a certain part of the ammonia formed reacts with terephthalic acid, the pressure begins to gradually decrease. The catalyst used in the reaction significantly increased the reaction of ammonia formed from the decomposition of urea with the OH functional group in terephthalic acid, which can be explained by the increase in reaction yield. The obtained product was washed in hot water at a temperature of 90 °C. In this way, it is purified from biuret and isocyanides formed during the reaction. After filtration, it is dried. Terephthalamide was obtained in 60-65% yield without a catalyst and 75-80% with the catalyst.

2.4. Chlorination of t Terephthalamide at low temperature and in a neutral environment. To start the reaction, 30 g of dry and powdered terephthalamide are placed in a 1-liter three-necked flask. During the passage of chlorine gas through the solution, the solution medium changes from alkaline to acidic. If the acidity level in the solution increases, the efficiency of the reaction of chlorine with terephthalamide decreases. Therefore, 100 ml of a 20% calcium hydroxide solution is added every 30 minutes. The reaction is carried out at a temperature of +5 °C to 0 °C. The reaction is carried out at a low temperature so that the calcium hydroxide used in the reaction does not react with terephthalamide and form organometallic salts. The process is carried out by mixing using a special mixer. At a constant rate of chlorine gas, a chlorine stream is passed through the colloidal solution at a rate of 4 g/min for 3.5 hours [26-27].

Chlorinated terephthalamide precipitated because it was insoluble in water. The solution was filtered and the precipitate remaining on the filter was dried. The calcium chloride was washed in water at 70°C to remove the salt and dried again at 40°C.

2.5. Synthesis of p-phenylenediamine from N,N'-Dichloroterephthalamide (TDAX). Hofmann's rearrangement reaction was used to carry out the synthesis process. For this, 200 ml of 10% sodium hydroxide solution was placed in a 500 ml heat-resistant beaker and cooled to +5 °C. At the same temperature, 20 g of TDAX in the form of a dry powder was added to the solution. The mixture of TDAX and sodium hydroxide solution was kept at +5 °C for 1 hour. After that, the mixture in the container was kept at room temperature for half an hour and then heated at 60 °C for 1 hour. During this time, a dark brown solution was formed. Then, with stirring, it was heated at 80 °C for another 1 hour. After the reaction was completed, an extraction method was used to purify the resulting mixture from substances such as NaCl, Na_2CO_3, and NaOH. Organic solvents such as toluene and benzene were used for extraction.

3. Results and discussion

3.1. Influence of various factors on the yield of terephthalamide (TPA)

The effect of the mass ratio of the initial substances. Fig. 2 shows a diagram that has the results of reactions carried out at 185ºC for 3 hours at different mass ratios of starting materials to achieve high yields. Since the amount of ammonia involved in the reaction was unknown and ammonia gas was removed from the reaction medium to control the pressure, the amount of urea obtained is much higher than the theoretically calculated amount. 2 g of NaCl (K1) was added as a catalyst to each reaction. From this diagram, it was found that the yield was higher when the mass ratio of terephthalic acid (TP) and urea (UR) was 1:1.4. The final result is *n*(TP):*n*(UR) = 1:1.4 and 1.6% catalyst (K1).

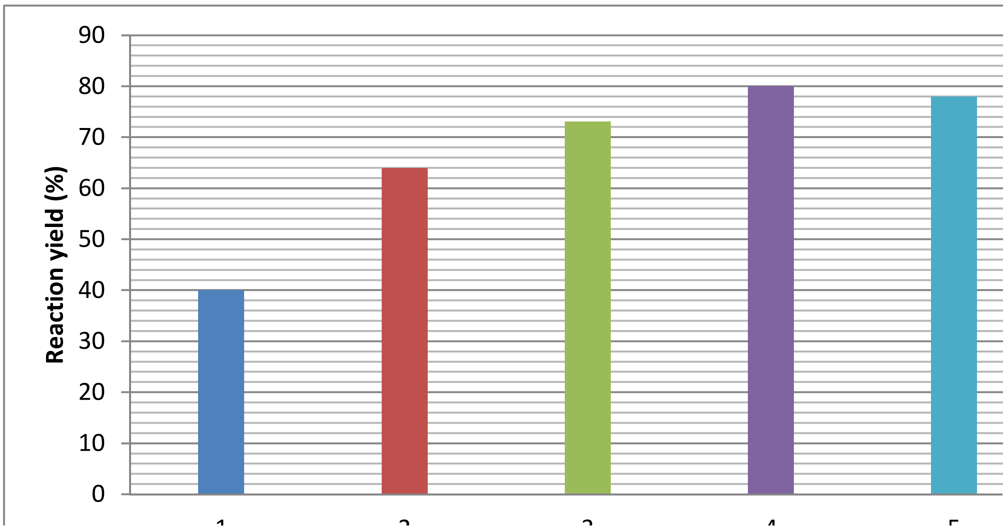


Fig. 2. Effect of different mass ratios of terephthalic acid and urea on the reaction yield

Effect of reaction temperature. The effect of different temperatures on the 3-hour reaction with a mass ratio of n(TP):n(UR)=1:1.4 and 1.6% catalyst (K1) was studied. The results obtained are presented in the graph in Fig. 3. From this graph, we can see that the reaction yield increases with increasing temperature, and the highest yield is obtained at 185°C and then decreases starting from 195°C. At temperatures up to 150°C, a certain part of urea is converted to biuret and begins to decompose at above 150°C and increases with increasing temperature. However, at temperatures above 185°C, the instability of the formed amides begins to increase, so 185°C was chosen as the optimal choice.

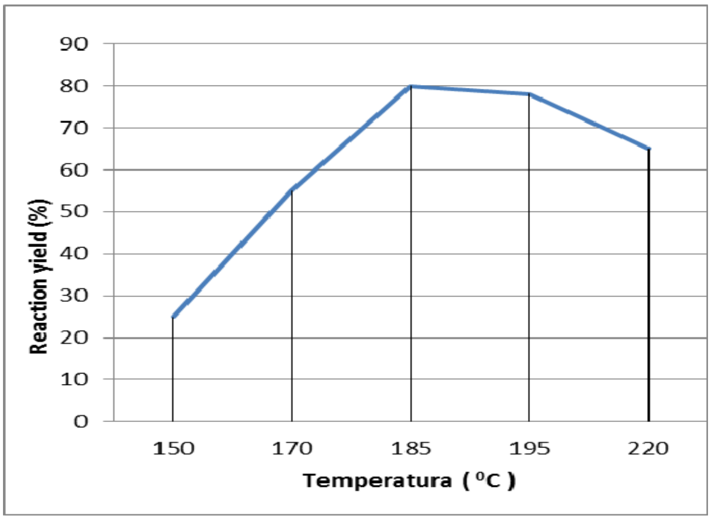


Fig. 3. Effect of temperature on the yield of the TPA production reaction

Effect of reaction pressure. The effect of different pressures on the reaction was studied at 185ºC, 1.6% catalyst (K1), at a mass ratio of n(TP):n(UR) = 1:1.4 and a reaction time of 3 hours. The results are presented in Fig. 4. It is shown that a high yield is achieved at a pressure of 6 atm, since the reactivity of ammonia gas released from urea decreases with increasing pressure. This situation occurs at pressures above 6 atm: the exchange reaction of ammonia gas formed at high pressure with the carboxyl functional group -OH in terephthalic acid decreases, which may be due to the decomposition of water vapor and ammonia. At low pressure (3-4 atm), ammonia gas does not reach sufficient activity to react. According to the results of the study, a pressure of 6 atm was chosen as the best option.

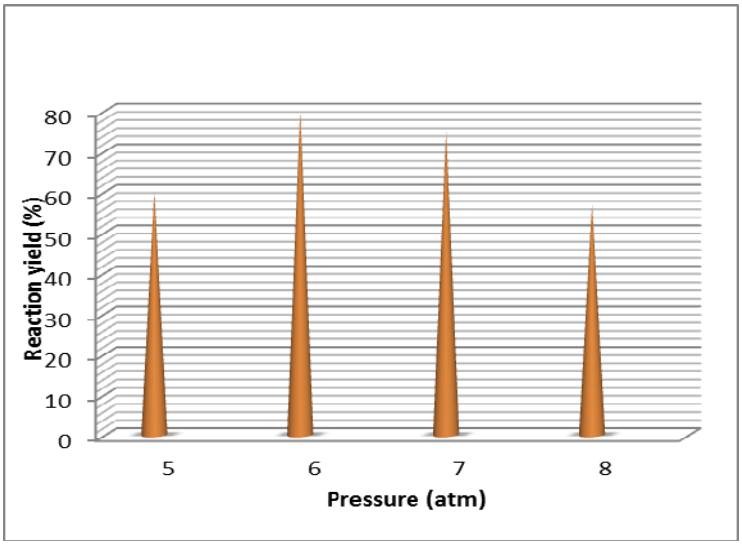


Fig. 4. Effect of pressure on the yield of the TPA production reaction

The effect of reaction time. The effect of reaction time was studied in 1.6% catalyst (K1), 185ºC, and a mass ratio of n(TP):n(UR) = 1:1.4 of the reaction. The results are shown in the diagram in Fig. 5. The results show that after 3 hours the reaction yield decreases. During 3 hours, ammonia and carbon dioxide are released from urea under the influence of temperature, as a result of which these two gases and the amount of water produced in the reaction system increase with time. This ammonia begins to have a significant effect on the reaction yield. The mixture of gases and water produced causes the decomposition of amides formed at high pressure. During the period up to 3 hours, ammonia reacts with the carboxyl functional group of the TP, and after this time, the decomposition reaction rate begins to increase. Therefore, the reaction time was set to 3 hours.

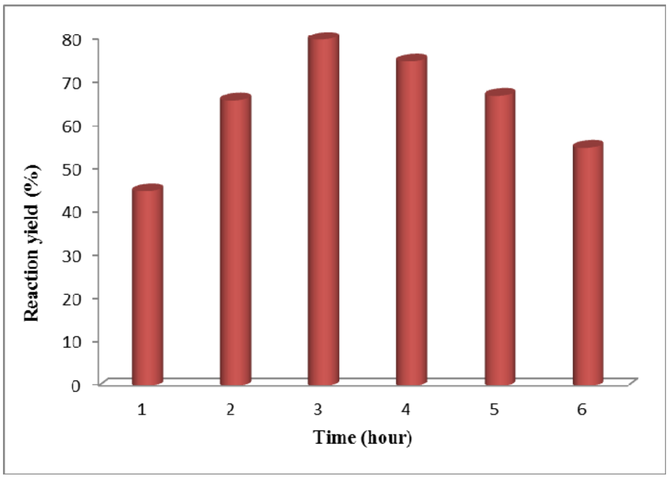


Fig. 5. Effect of time on the yield of the TPA production reaction

Effect of the catalyst. The effect of different catalysts was studied in the reaction of 1.6% catalyst (K1), 185ºC, 3 hours, and a mass ratio of n(TP):n(UR) = 1:1.4. Fig. 6 shows the results of the study of the effect of inorganic salts used as catalysts on the reaction yield. Various salts were tested as catalysts in many reactions, and the results of the best performers among them are presented in the table. $CuCl_2$, NaCl, $Ca(OH)_2$, and $MgSO_4$ were able to affect the reaction yield when used as catalysts. Among these, the highest result was obtained when NaCl was used. However, the recovery of the used catalysts for reuse after the reaction leads to a very complicated process. Therefore, attention was paid to economically efficient types when choosing a catalyst. The catalyst increased the degree of reaction of the carboxyl functional group in terephthalic acid with ammonia gas and increased the reaction yield. Using the obtained results, NaCl was selected as the catalyst.

In the diagram, it can be observed that $CuCl_2$ also produced favorable results. Both $CuCl_2$ and monovalent copper salts are generally considered effective catalysts. However, in this reaction, the use of monovalent copper salts did not yield satisfactory results. Their excessive catalytic activity accelerated side reactions, ultimately reducing the overall reaction yield.

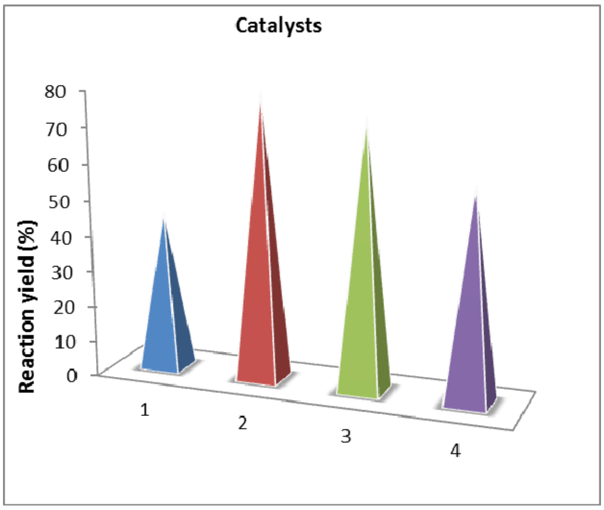


Fig. 6. Effect of catalysts on the yield of the TPA production reaction: 1 - $CuCl_2$, 2 – NaCl, 3 - $Ca(OH)_2$, 4 - $MgSO_4$

3.2. The influence of various factors on the chlorination yield of terephthalamide.

Effect of chlorine flow. The reaction yield was measured at different chlorine flow rates and time intervals through a colloidal solution of terephthalamide prepared with water. Table 1 was compiled based on the results obtained.

Table 1. Effect of chlorine flow rate on chlorination reaction yield at different time intervals

Chlorine flow (gr/min)	Time (hours)	Reaction yield (%)
1	1	45
2	1,5	60
3	2	65
3,5	3	78
4	3,5	95
4,5	4	94

High yields cannot be achieved with this colloidal solution at short times and low chlorine flow rates. Experiments conducted at high chlorine flow rates and for short times ended with low yields, and experiments were conducted in a calculated sequence to determine the optimal time and chlorine flow rate. High yields were achieved when chlorine flow was passed through the colloidal solution at a rate of 4 g/min for 3.5 hours, and the yield remained almost unchanged in subsequent reactions.

The influence of the reaction environment. Fig. 7 shows the results of studying the effect of the environment on the yield of the chlorination reaction of TDA. During the chlorination process, chlorine passing through the colloidal solution dissolves in water, causing the environment to become highly acidic. In this environment, the reaction of amides with chlorine becomes difficult, and a product is obtained with low yield. Therefore, we can see that the yield is low when the pH is -1, 2, 3, or 4. As the environment changes from neutral to alkaline, the degree of reaction of chlorine passing through the solution with calcium hydroxide increases, and as a result, the chlorination reaction of amides decreases and the reaction yield begins to decrease. Chlorination processes were carried out in solutions with different pH values, and the reaction yields at each stage were calculated. According to the results obtained, the highest reaction yield was achieved in a neutral environment.

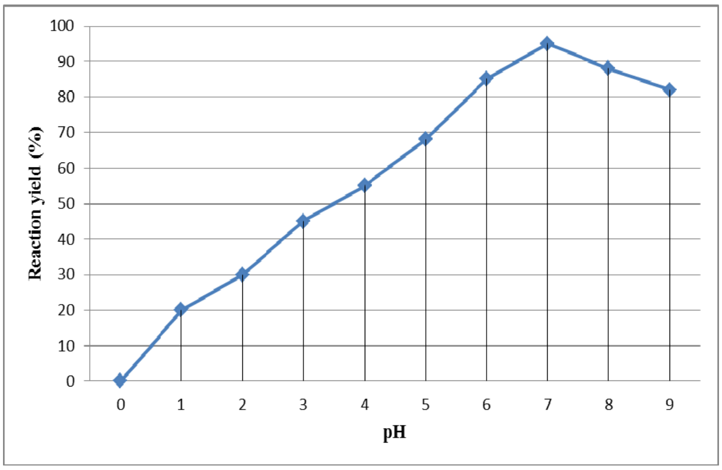


Fig. 7. Effect of the chlorination reaction on the environment

Effect of temperature on chlorination reaction yield. In the process, it was found that chlorine is mainly more active at low temperatures. Experiments conducted at high temperatures did not give good results. Fig. 8 shows a graph based on the results obtained when chlorination was carried out at different temperatures. At temperatures below 5ºC, the reaction yield decreases. This is because the solubility of calcium hydroxide added to the colloidal solution for neutralization decreases, and it begins to precipitate. The most optimal temperature was found to be 5-6ºC, according to the analysis of the results obtained.

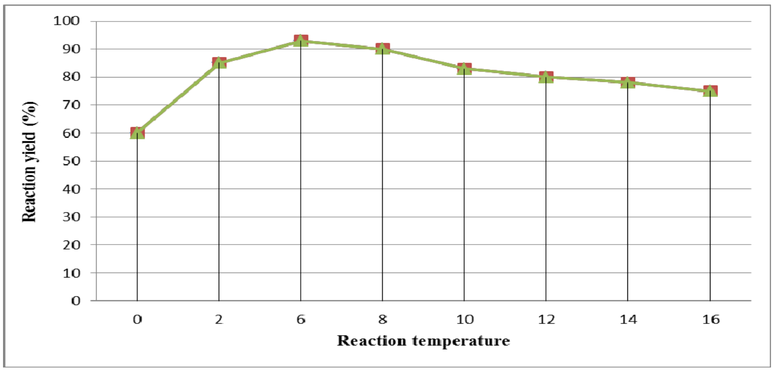


Fig. 8. Effect of temperature (ºC) on the yield of the chlorination reaction

3.3. SEM analysis. A sample of the synthesized terephthalamide was taken and analyzed by using a MIRA 2 LMU scanning electron microscope (Fig.s 9, 10, and 11). Microanalysis of the elements in the obtained sample was carried out and studied in areas with an accelerating voltage of 20 keV and a current of 1 nA. An SEM image of the obtained sample was presented, and the degree of reaction and the state of the porous layers were described. Along with SEM, the mass fractions of the elements in the sample were also determined (Fig. 11).

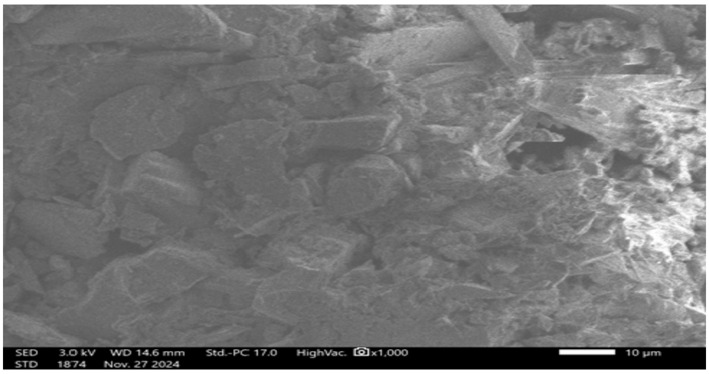


Fig. 9. Scanning electron microscope image of synthesized terephthalamide

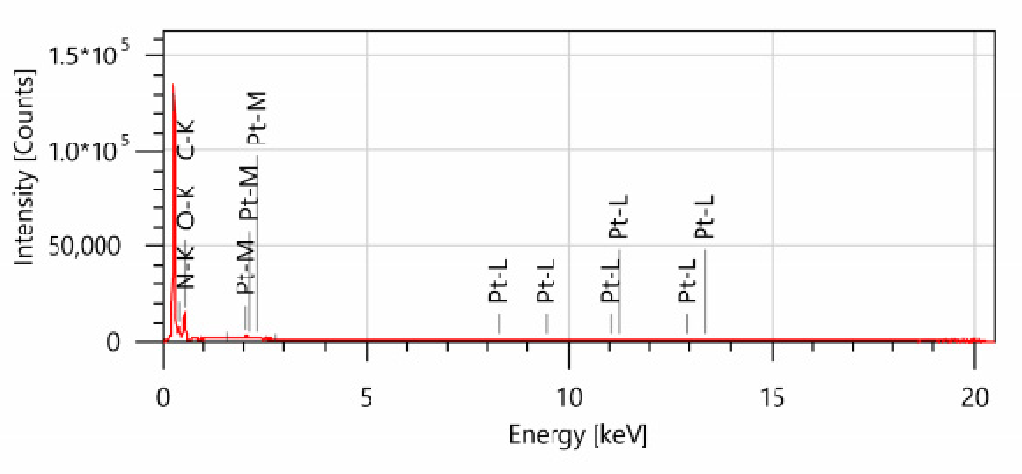
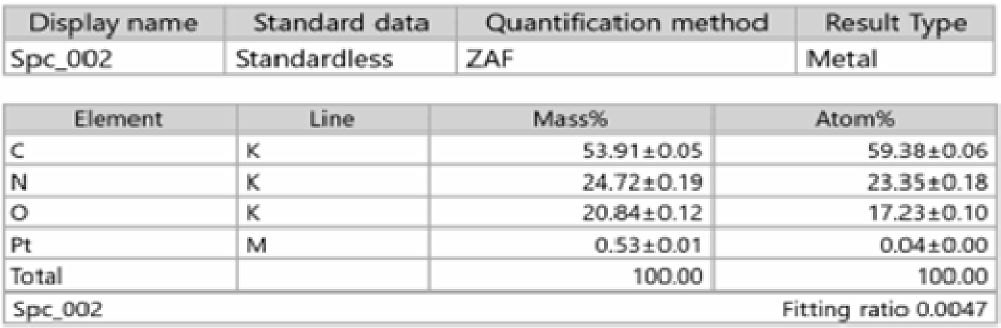


Fig. 10. Elemental analysis of synthesized terephthalamide



Display name	Standard data	Quantification method	Result Type
Spc_002	Standardless	ZAF	Metal

Element	Line	Mass%	Atom%
C	K	53.91±0.05	59.38±0.06
N	K	24.72±0.19	23.35±0.18
O	K	20.84±0.12	17.23±0.10
Pt	M	0.53±0.01	0.04±0.00
Total		100.00	100.00
Spc_002			Fitting ratio 0.0047

Fig. 11. Elemental analysis of synthesized terephthalamide obtained by SEM

3.4. IR Spectrum of Analysis. The asymmetric and symmetric stretching vibrations of the NH_2 groups in the structure of p-phenylenediamine can be observed in the doublet state at 3373 cm^{-1}, 3307 cm^{-1}, and 3197 cm^{-1}, and the deformation vibrations of this group can be observed at 1627 cm^{-1} and 1514 cm^{-1}. The stretching vibrations of the C-N bond are observed at 1259 cm^{-1}, and there are bands in the spectrum at 3007-2929 cm^{-1} due to the stretching vibrations of the C-H bonds in the aromatic ring. In the regions of 1444 cm^{-1} and 1340 cm^{-1}, the stretching vibrations of the aromatic ring along the ring plane can be observed (Fig. 12).

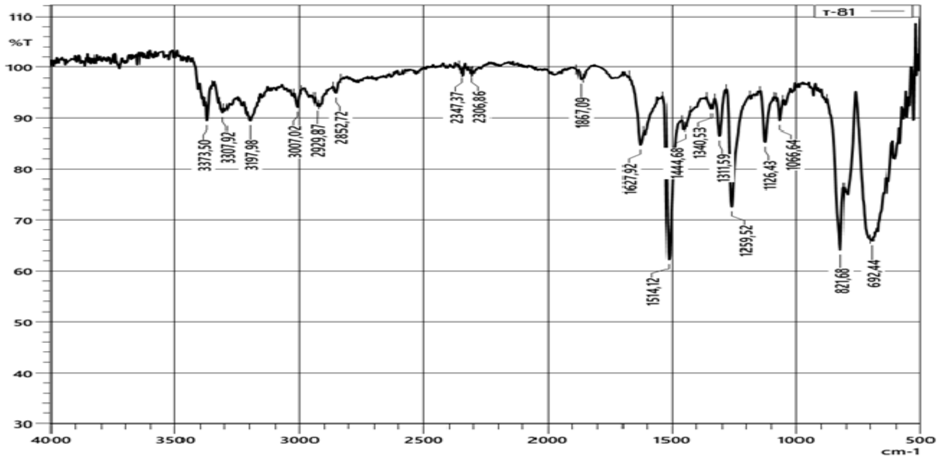


Fig. 12. IR spectrum of the resulting p-phenylenediamide

3.5. Differential-thermal and Thermogravimetric Analysis. DTA analysis of the resulting p-phenylenediamine showed three exothermic peaks at 163.14°C, 241.56°C, and 537.13°C. No endothermic process was observed. The exothermic peaks at 163.14°C and 241.56°C were due to the decomposition of amines (NH_2). The energy released for these exothermic processes was 17.24 mcal (72.16 mJ) and 26.66 mcal (111.59 mJ), respectively. The exothermic peak at 537.13°C was observed to be due to the decomposition of the benzene ring or the breaking of covalent bonds. The energy released for this exothermic process was 304.29 mcal (1.26 J).

According to the TGA results of the obtained p-phenylenediamine, the substance decomposed in two stages. The first stage occurred between 12.58°C and 360.65°C and lost 34.14% of the total mass (1.172 mg). The second stage occurred between 360.65°C and 801.73°C, and 46.84% of the total mass (1.608 mg) was decomposed under the influence of temperature (Fig. 13).

Table 2. TGA analysis of the resulting p-phenylenediamine

Reaction process	Reaction process duration	Temperature range	Mass loss (%)
Stage 1	36.48 min	12.58°C-360.65°C	34.14 %
Stage 2	2007 sec	360.65°C-801.7°C	46.84%

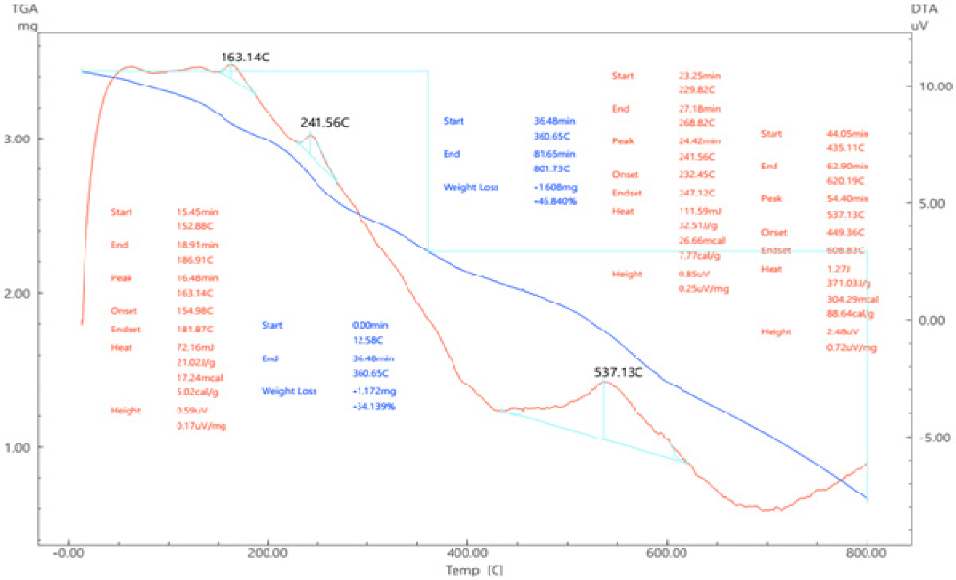


Fig. 13. TG and DTA analysis results of the obtained p-phenylenediamine

Conclusion

Terephthalic acid from PET waste was catalytically aminated and chlorinated in a neutral medium, and p-phenylenediamine was obtained in high yield through a Hoffmann rearrangement reaction. The effect of various factors on each reaction step was studied. In the amidation reaction of terephthalic acid, the mass ratio of terephthalic acid and urea was 1:1.4, the reaction temperature was 185°C, the pressure was 6 atm, 1.6% (K1) catalyst, and 3 hours were found to be the most optimal option. In the chlorination reaction of TDA, the most optimal option was found to be the chlorine flow rate of 4 g/min, 5°C, pH 7, and 3.5 hours.

The intermediate product terephthalamide obtained during the reaction steps was examined using SAM and elemental analyses to increase the reaction yield. At the end of the processes, a product with a yield of 95% was obtained, and its properties were examined using TG, DTA, and IR-spectrometry analyses and proved to be compatible with P-phenylenediamine.

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