

A. H. ABBAR^{1*}, A. H. SULAYMON²
and M. G. JALHOOM³

Scientifica paper
UDC:661.717.3:621.357.3=20

A Novel, Pilot Scale Electrolysis System for Production of P-Aminophenol using Parallel Screen Electrode

The present research investigates the nitrobenzene reduction to p-aminophenol by using a novel pilot scale electrolysis system consisting of parallel amalgamated copper screens electrode followed by direct acetylating to paracetamol. The preparative electrolysis of nitrobenzene was carried out using supporting electrolytes consisting of (2M) H₂SO₄ in a solution of 50% 2-propanol/50%water (v/v) and two modes of operation galvanostatic (controlled current) and potentiostatic (controlled potential). A Factorial experimental design was adopted for studying the effect of electrode potential, nitrobenzene concentration, flow rate, and number of screens in the electrode on the yield of p-aminophenol.

The results indicated that yield of p-aminophenol increases with cathode potential more positive than (-400mV vs. SCE), increasing flow rate, decreasing nitrobenzene concentration, and number of screens. The best results were obtained at cathode potential (-375mV vs. SCE), catholyte flow rate (111cm³s⁻¹), nitrobenzene concentration (0.1gcm⁻³) and number of screens not grater than five in which p-aminophenol yield was (80%), with maximum conversion (83%) of p-aminophenol to paracetamol.

Key words: Nitrobenzene, P-aminophenol, electrochemical reduction, Preparative electrolysis, Screens electrode.

1. INTRODUCTION

P-Aminophenol is a commercially important chemical intermediate used in the manufacture of several analgesic and antipyretic such as paracetamol, acetanilide, and phenacetin etc. Apart from pharmaceuticals, p-aminophenol is also used in the manufacture of various industrial dyes such as sulfur and azo dyes, which are especially useful in dyeing hair, furs and feathers [1]. P-Aminophenol is a strong reducing agent and is also marketed as a photographic developer under the trade names of Activol, Azol, Certinal, Citol, Paranol, Rodinol, Unal, and Ursol-P either alone or in combination with hydroquinone. The oxalate salt of p-aminophenol marketed under a trade name of Kodelon is used as a corrosion inhibitor in paints and anticorrosion – lubricating agent in two – cycle engine fuels. Another use of p-aminophenol is in the production of rubber antioxidants [2].

Because of its industrial importance, synthesis of p-aminophenol has attracted considerable interest. It is manufactured chemically by several methods: (i) one method consists of the reduction of p-nitrophenol

with iron and HCl, which causes waste disposal problems; (ii) phenol may be treated with HNO₃, which on reduction with poly – sulfide, gives p-aminophenol with very poor yield; (iii) nitrobenzene may be reduced to p-aminophenol in the presence of catalysts such as Pt or Pd and this method is highly expensive. The recovery of the catalysts used is a significant economic factor in this process [3]. An alternative method for production of p-aminophenol is by the electrolytic method, which is clean and without pollution problems. The yield is higher in comparison with the chemical method, leading to purer product. The electro reduction of p-nitrophenol to p-aminophenol involves 6 Fmol⁻¹ whereas the electro reduction of p-nitrosophenol or nitrobenzene involves only 4 Fmol⁻¹. The maximum yield of p-aminophenol from p-nitrosophenol was only (78%) while a higher yield was obtained from nitro benzene [4].

The electro reduction of nitrobenzene in acidic medium has been well studied, and known for nearly a century. A simplified version of the reduction sequence is shown in figure 1[5]. The reduction of nitrobenzene involves the transfer of two electrons to form nitrosobenzene. The nitrosobenzene is then further reduced to phenyl hydroxyl amine with a further transfer of two electrons. Finally, with the transfer of two more electrons the phenyl hydroxyl amine formed is reduced to aniline.

Address authors: ¹QADESSYIA University, Department of Mechanical Engineering, Iraq, ²Baghdad University, Environmental Engineering Department, Iraq, ³Ministry of industry, Ibsina State Company, Iraq, (*author for correspondence, e-mail: aliha68@yahoo.com)

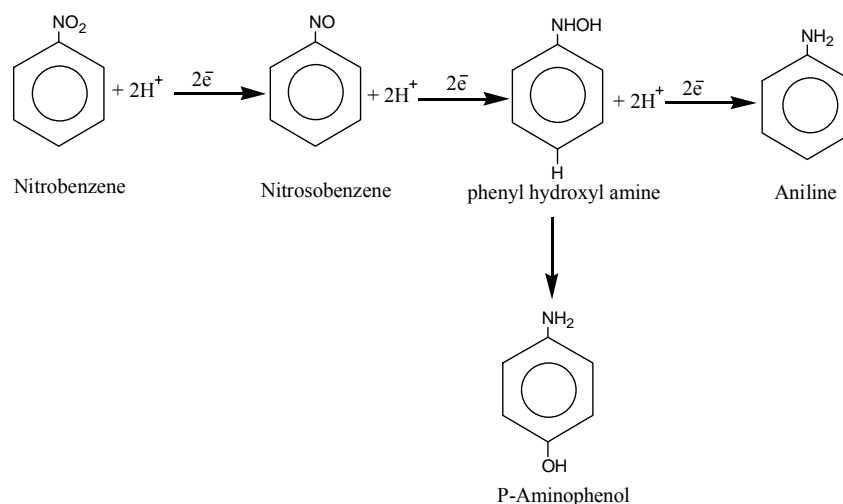


Fig.1 - The mechanism of p-aminophenol preparation by electrolytic method

If it is desired to form predominantly p-aminophenol by rearrangement of phenyl hydroxyl amine, an acid medium is required and precise potentiostat control potential is important in order to obtain the desired reaction product. The electrolytic reduction of nitrobenzene to p-aminophenol was first described by Gattermann and Kopper[6], using a platinum cathode at an ambient temperature in a highly acidic reaction medium. Haber [7] published a paper dealing with the step – wise electrolytic reduction of nitrobenzene at the cathode. In this paper, Haber pointed out that, by controlled variation of the cathode potential, either p-aminophenol or aniline could be obtained in predominant yield as the products of the reduction reaction occurring at the cathode. From this, Haber concluded that in such step – wise organic electrochemical reduction, the most important single factor in controlling the progression of the reaction and the degree of reduction was cathode potential.

Many factors that influenced the cathode reduction of nitrobenzene have been studied in previous works[8-19], these are cathode potential, cathode material, solvent, PH, concentration, temperature, and agitation. The studies performed were mainly concerned with the effect of these factors on the conversion, yield and selectivity of p-aminophenol. The cathode forms used in the literatures were cylinders; bars, perforated sheets or plates. The production rate using these configurations was low because of the lower specific surface area of these types of cathode. Only Lawson[20], Goodridge and Hamilton[21], and Delue [22] used fixed bed electrode for the preparation p-aminophenol on laboratory scale. This type of electrodes gives higher specific surface area than the traditional types. Lawson prepared p-aminophenol by

electroreduction of nitrobenzene using a fixed bed cathode consisting of Monel particles with size range (200 μm to 1000 μm). Goodridge and Hamilton studied the behavior of a porous flow – through electrode during the production of p-aminophenol. They used particles made of Monel (400 alloy) and catholyte containing molar sulfuric acid, they found that the behavior of porous flow through electrode (PFTE) for a reaction was essentially activated controlled regime and satisfactorily explained by a simple one – dimensional model, provided concentration changes per cell pass and in the direction of current flow was negligible. Delue used copper and amalgamated copper screen cathode for the preparation of p-aminophenol by electroreduction of nitrobenzene using catholyte consisting of(68%) distilled water, (24%) sulfuric acid, (0.12 %) dim ethyl – dodecyl amine – N-oxide, and(7%) nitrobenzene. He obtained p-aminophenol to aniline molar ratio of a bout (22 to 1).

Recently Polat[23,24], studied the electroreduction of nitrobenzene to p-aminophenol on a scale higher than laboratory scale, firstly, they used voltammetric and semi pilot scale preparative electrolysis techniques where copper and amalgamated copper plate electrodes in addition to catholyte consisting of (2M) H₂SO₄ in a solution of 50% 2-propanol /50% water (v/v) have been used. They deduced that most suitable electrode material was amalgamated copper and the reduction of nitrobenzene to p-aminophenol was found to follow an electrochemical-chemical – electrochemical mechanism. Secondly, they used a novel, semi – pilot scale electrolysis system for the production of p-aminophenol using TiO₂ / Ti, Sn and Pb electrodes but the maximum yield of p-aminophenol was (60%).

The aim of the present research is to study the effect of the main parameters of the nitrobenzene electro reduction on the yield of p-aminophenol using pilot scale system consisting of parallel screens electrode made from amalgamated copper and catholyte consisting of (2M) H₂SO₄ in 50% 2-propanol /50% water (v/v).

The second aim of the present work is the direct inexpensive acetylation of the electrolytically produced of p-aminophenol to paracetamol where, in converting the p-aminophenol in the electrolytic reduction mixture to paracetamol, the initial neutralization of the considerable excess of the mineral acid which is usually present, requires the use of equimolar amount of sodium hydroxide in the quantities which are necessary to effort the desired neutralization .The sodium hydroxide employed represents a major factor in the overall production cost .furthermore, the isolation and purification of the p-aminophenol obtained as a crude reaction product also contributes materially to the cost ,therefore calcium carbonate will be used instead of sodium hydroxide. This material eliminates the costly use of sodium hydroxide as a neutralizing agent and also eliminates the necessity for separating p-aminophenol prior to acetylation.

2. EXPERIMENTAL

2.1. Characterization and preparation of amalgamated copper electrode

Copper circular screen cathodes with (0.19mm) diameter wires woven at 50 per inch were used. The wire diameter was measured by the electronic length indicator model (TESA/ Germany made). The cross section area of each screen was (227cm²) its thickness was (0.4mm). The specific surface area(S) and void fraction (ϵ) were calculated according to the following correlations [25]:

$$S = \frac{\omega Nr}{L\rho_{cu}} \quad \epsilon = 1 - \frac{\omega N}{L\rho_{cu}}$$

Where ω is the average weight area density of single screen (0.065gcm⁻² measured value).N number of screens in the electrode, ρ_{cu} density of copper (8.47 gcm⁻³ measured value)and, r is a ratio of surface area to volume of wire(d) equal to 4/d. For d= 0.019cm, r=210.5cm⁻¹, L is thickness of screen .The values of L, S , ϵ and the ratio of actual surface area to the cross sectional area for the applied electrode(A_a/A) are given in table 1.

It is clear that the ratio of actual area to cross section area of electrode increases rapidly with increasing thickness of bed, however this will be limited by the penetration length of the electrochemical

reaction through the bed which controlled by potential distribution.

Table1 - Properties of the screen

Number of screens	L Thickness(cm)	S (specific surface area) cm ⁻¹	ϵ (void fraction)	A _a /A
1	0.040	40.39	0.808	1.617
2	0.090	35.89	0.829	3.230
3	0.130	37.20	0.823	4.836
4	0.175	36.84	0.824	6.447
5	0.220	36.71	0.825	8.070

The copper screen cathode was first treated with dichloromethane in order to remove any grease from the electrode surface then with cleaning solution consisting of H₂SO₄ (0.1gcm⁻³) and sodium dichromate (0.06gcm⁻³), followed by dipping in neutralization solution of sodium carbonate (0.025gcm⁻³) at 25°C, shaken in distilled water for a few minutes and wiped with drying paper before being placed in the amalgamation solution. The copper screen cathode was amalgamated by coating mercury using electrolyzing solution of 1%mercuric sulphate, with a copper cathode and lead anode at a current density of 5 Adm⁻² for 15 min.

Cell set-up and electrolysis

A pilot scale electrolysis cell with overall dimensions 25cm height×25width×17cm length was used. It consisted of two main parts: cathode chamber and anode chamber separated by an ionic ion –exchange membrane (MA-3475). The cathode chamber was made of Teflon with outer dimensions 25cm height×25width×12cm length containing three elements: copper current feeder, amalgamated copper screens, and calming screens made from Teflon net. The cathode compartment was divided into three sections: the first for the distribution of catholyte at inlet, the second was for fixing calming screens, amalgamated cathode screens, and current feeder, the third was for collecting the catholyte solution for output. The compartment was provided with openings for input the capillary loggin tube and electrical connections. The anode chamber was made also from Teflon with outer dimensions 25 cm height×25width×5cm length containing disk of graphite as anode with dimensions 16cm diameter×2cm thickness. Figure 2 shows the details of electrolytic cell.

The pilot flow system used in the present work consisted of anode reservoir, cathode reservoir, pumps, two plate heat exchangers, and electrolysis cell. The reservoirs were made of rectangular porcelain tanks with capacity of twenty liters each of them provided with PVDF covers. The pumps were mag-

netic centrifugal with PVDF heads, maximum flow ($500\text{cm}^3\text{s}^{-1}$). Plate heat exchanger made from PVDF frame and titanium plates with outer dimensions 30cm length \times 30 width \times 16cm height Circulating heat-

ing system (LOUDA/Germany) was used to heat the catholyte and anolyte solutions through plate heat exchangers. Figure 3 shows the pilot plant system.

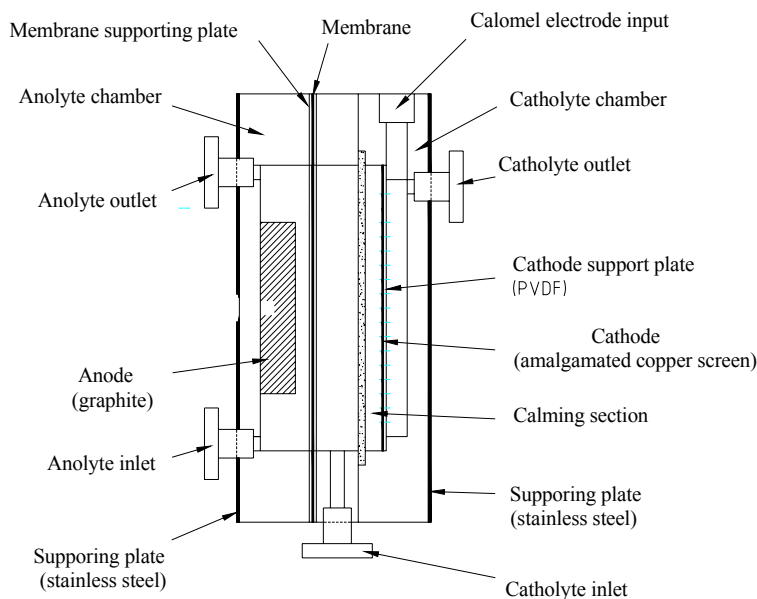


Fig. 2. The electrolytic cell

All the reagents used in the experiments were analytical grade (Merck) and used without further purification except nitrobenzene which distilled at 220°C to remove any possible contaminants. Distilled water with a conductivity of $1/1.3 \times 10^{-6} \Omega^{-1}\text{cm}^{-1}$ was used throughout the experiments. The catholyte used was (2M) H_2SO_4 in a solution of 50% 2-propanol /50%water (v/v) while the anolyte was only (2.M) solution of H_2SO_4 . Nitrobenzene was added to the catholyte at concentration range (0.1gcm^{-3} - 0.25gcm^{-3}) two types of operation mode were used: potentiostatic and galvanostatic the first mode of operation achieved by using potentiostat model (LARSA-potent-50A/50V/Iraq), while the second mode of operation achieved by using power supply model (LARSA -PS/V-50/50/Iraq) with output current and voltage (50A, 50V) respectively. Before starting each run fifteen liters of each catholyte and anolyte were added to their reservoirs, the pumps were switched on for circulating the catholyte and anolyte in the system through the by pass sections without letting them to flow through the electrolytic cell, the nitrobenzene was then added to the catholyte with the appropriate concentration. Nitrogen gas was bubbled through the catholyte for one hour. After the temperature of two electrolytes reached 70°C the electric current leads were connected to the electrolytic cell and reference electrode. After checking all the electrical connec-

tions, the electrolytes were allowed to flow through the electrolytic cell by closing the by pass valves and opening the electrolytic cell valves. The polarization curve for each run was recorded and the operation was continued for 8hr at the required control potential or current. At the end of each run the power supply or potentiostat were switched off, the drain valves opened in order to drain the used electrolytes. The pilot system was than washed entirely and especially the electrolytic cell by using distilled water to make sure that there was no electrolytes left in the system. The electrolytic cell was dismantled to replace the used cathode with a new one. Samples of catholyte were taken and analyzed by high performance liquid chromatography to determine the percent of p-aminophenol and aniline.

2.3. Separation of the Final Product

The catholyte obtained from electrolytic reduction of nitrobenzene at the preferred conditions was distilled to remove isopropanol then neutralized with calcium carbonate to pH 4.5. The precipitated calcium sulfate is separated by filtration, the filtrate extracted twice with toluene then the aqua's phase separated and treated with activated carbon to remove any organic contaminants. The required amount of anhydrous sodium acetate and acetic anhydride was added to the aqua's phase at 40°C , cooled to ($8 - 10^\circ\text{C}$) for separation of paracetamol.

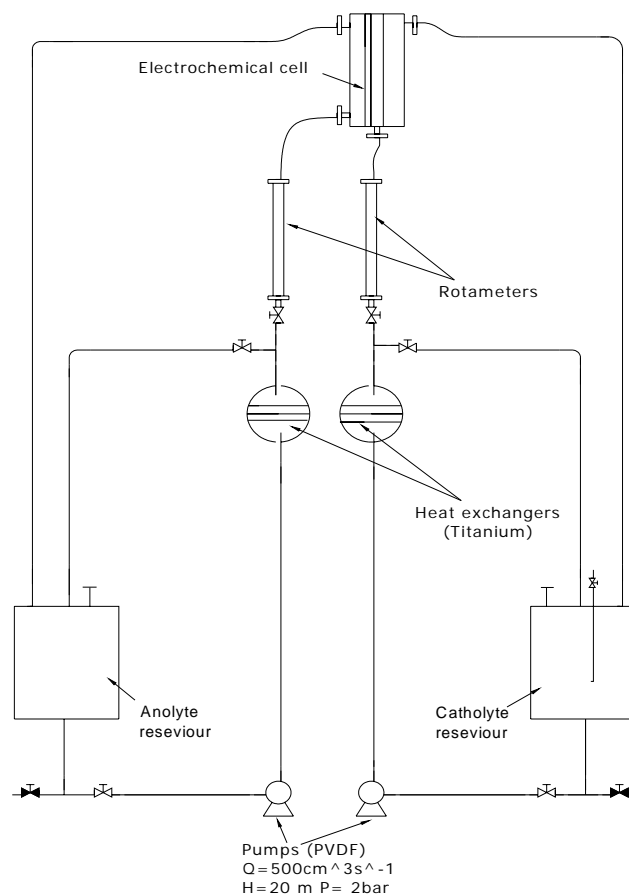


Fig. 3. The electrochemical systems for preparation p-aminophenol

3. RESULTS AND DISCUSSION

3.1. Polarization curves

The polarization curves for the reduction of nitrobenzene on a fixed bed electrochemical reactor consisting of amalgamated copper screens electrode were obtained at different conditions. The general form of the current/potential curve for the system is represented in figure 4 as a plot of $\log(\text{current})$ against cathode potential (vs. Standard Calomel Electrode (SCE)). the general curve obtained may be explained as follows:

The reactions that take place at cathode surface initially are the reduction of nitrobenzene, or the intermediate, phenyl hydroxyl amine, and the production of gaseous hydrogen. At potential greater than that required for reduction of nitrobenzene (-200 mV) the current increases rapidly with increasing negative potential (AB). Over the potential range -400 to -600 mV the complete reduction of nitrobenzene to aniline proceeds with increasing rate where the mass

transfer effect observed (BC). At electrode potential higher than (-600 mV) hydrogen gas is evolved with increasing rate. Since the present work was involved with the reduction of nitrobenzene to p-aminophenol rather than aniline. The initial part of the curve (AB) is the most importance and will be considered in the next analysis of the present work. The current/potential curves for the effect of flow rate, nitrobenzene concentration, and screens number were plotted as $\log(\text{current})$ vs. cathode potential within the range (-250 mV to -425 mV) as shown in figures (5-7). The plots are linear in almost conditions, this linearization indicates that the reaction is electrochemical-chemical-electrochemical (ECE) type and diffusion controlled not mass controlled. Only at flow rate ($27.8 \text{ cm}^3 \text{ s}^{-1}$) the effect of mass transfer observed at potential greater than (-300 mV). This behavior can be interrupted by the fact that the intermediates remain at the surface of the electrode for a much longer period, which increase their chance of reaction and reconverting to aniline. The previous work results

[23] substantiated this characteristic. Therefore it is recommended to operate at flow rate higher than ($27.8\text{cm}^3\text{s}^{-1}$) to overcome the completely conversion of nitrobenzene to aniline.

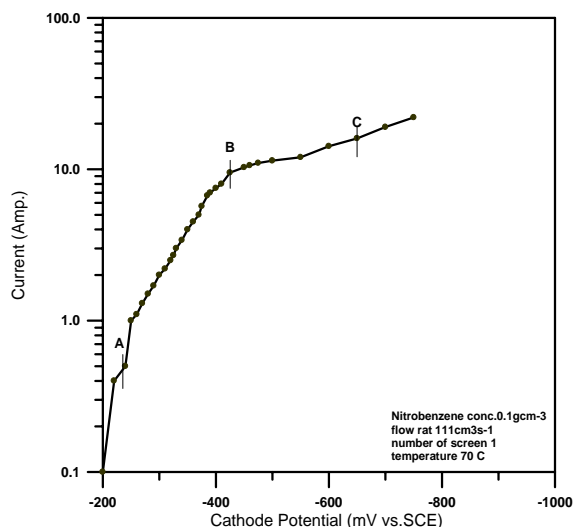


Fig. 4. The general polarization curve

From figure 5 one can noticed that at constant potential, the current increases with increasing electrolyte flow rate (i.e. the cathode become less polarized). As increasing electrolyte flow rate, the diffusion boundary layer near the surface becomes thinner which permits higher current to be passed.

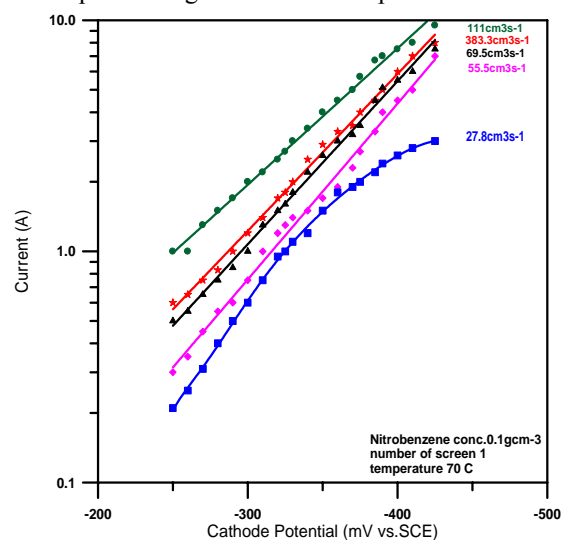


Fig. 5. The polarization curves at different flow rates of catholyte

The effect of nitrobenzene concentration on the current/potential curves (figure 6) is similar to that obtained for high flow rates. The current increases with increasing nitrobenzene concentration but with

less degree than flow rate effect. The effect of screen number on the polarization is shown in figure 7 where higher currents obtained as the number of screens increased due to increasing the surface area of the reaction. The increasing in current as the number of screens increased at constant potential is not linear, this behavior results from potential distribution in the bed, which makes some regions operated at lower potential and current than others.

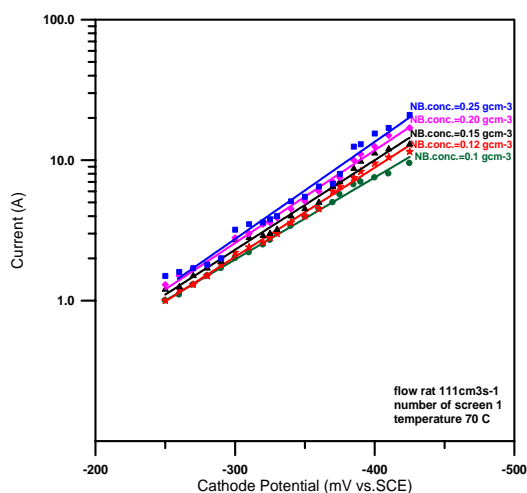


Fig. 6. The polarization curves at different nitrobenzene (NB.) concentration

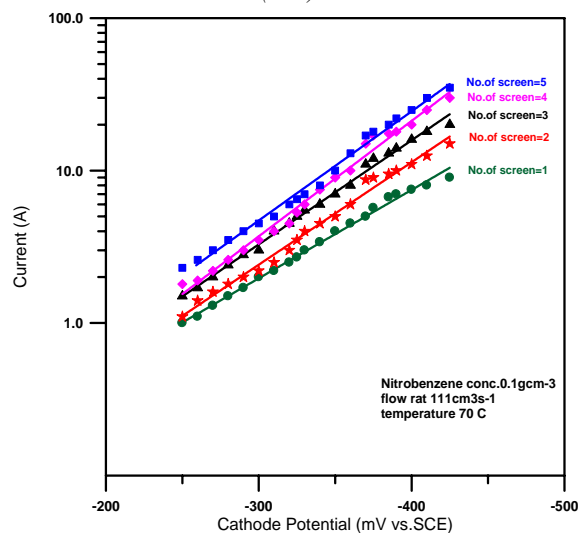


Fig 7. The polarization curves at different screens number

3.2. Potentiostatic operation

3.2.1. Effect of cathode potential

The results of series production runs made at various controlled cathode potentials are presented in figure 8 as plot of p-aminophenol yield against the

cathode potential. Although over the range of cathode potentials investigated both p-aminophenol and aniline are found, the further reduction of phenyl hydroxyl amine can be limited by maintaining the cathode potential more positive than (-375mV). A sharp decreasing in p-aminophenol yield occurred at potential more negative than (-400mV).

This suggests that the increased activity at the cathode surface enable the total reduction to aniline to proceed with increasing rate. Therefore it seems that electrode potential at (-375mV) be more suitable from practical point view because higher current with acceptable yield could be obtained. Polat [23] substantiated this characteristic. Lawson [20] in his work deduced that cathode potential (-300mVvs. SCE) is suitable to obtain higher yield on Monel fixed bed electrode. Polat[23] obtained yield 76.9% on planar amalgamated copper electrode at cathode potential equal to (-225mVvs. SCE). In the present work higher yield was obtained (85%) at potential electrode (-375mV) with maximum current.

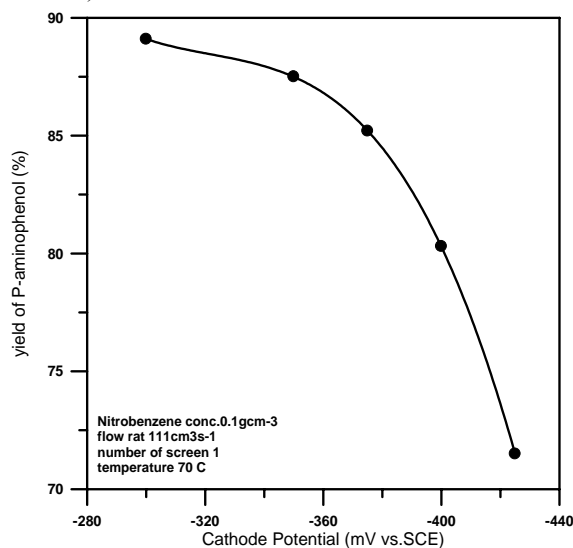


Fig. 8. The effect of cathode potential on the yield of p-aminophenol

3.2.2. Effect of flow rate

Figure 9 represents the effect of flow rate on the yield of p-aminophenol. The yield increases with increasing flow rate. Since the yield at flow rates in the range (83.3-111cm³ s⁻¹) are closer. It is not recommended to use flow rate higher than 111cm³ s⁻¹. The Reynolds number for flow through screens electrode can be expressed as follows[20]:-

$$Re = \frac{Qd}{\nu A}$$

Where Q is the flow rate of electrolyte, ν solution kinematics viscosity, ϵ void fraction, A cross section area of cathode bed, and d screen wire diameter. At the present work for nitrobenzene Concentration equal to 0.1gcm⁻³, Q =111 cm³s⁻¹, A =227 cm², d=0.019cm, $\epsilon=0.8255$, $\nu=0.5$ cm²s⁻¹. The value of Re in the present work was 0.56324. The previous works demonstrated that Re in the range 0.2-10 being more suitable for the reduction of p-aminophenol on fixed bed electrode [18-20].

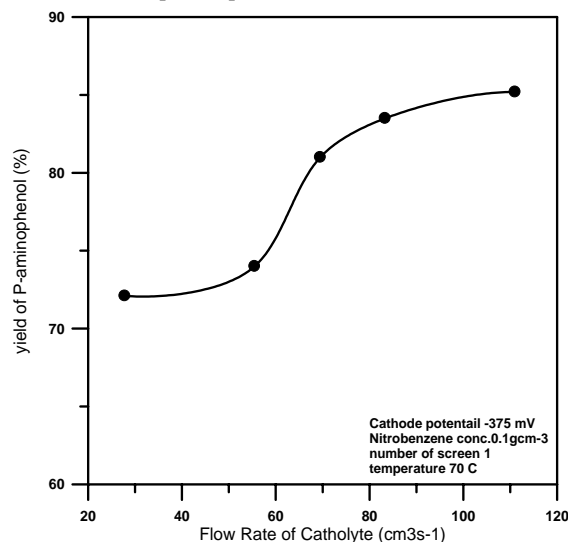


Fig. 9. The effect of catholyte flow rate on the yield of p-aminophenol

3.2.3. Effect of Nitrobenzene concentration

Figure 10 shows effect of nitrobenzene concentration on the yield of p-aminophenol. The increasing in nitrobenzene concentration led to decreasing p-aminophenol yield; this can be explained as follows: the increase in nitrobenzene concentration increases the chance of nitrobenzene and intermediate phenyl hydroxyl amine giving reaction at the cathode surface. This reaction is promoted as the amount of non dissolved nitrobenzene increases which occurred at nitrobenzene concentration higher than 0.2 gcm⁻³.

3.2.4. Effect of screens number

Figure 11 illustrates the effect of screens number on the yield of p-aminophenol, the yield decreases as screens number increased when the system operates at constant potential measured at the back of screens electrode. It was expected that the yield will decrease sharply when the number of screens electrode exceeded (5) in which the potential at front of electrode reach to hydrogen evolution potential. Therefore an additional experiment using eight screen electrodes

was performed at the same condition to emphasis this conclusion. A comparison between the current and yield obtained is shown in table 2 where a sharp decrease in the yield was obtained at number of screen equal to (8). Decreasing electrode potential at the back of electrode lower than (-375mV) leads to decrease the total current taken by the cell. Therefore using screen numbers equal to 5 being more suitable for reduction of p-aminophenol.

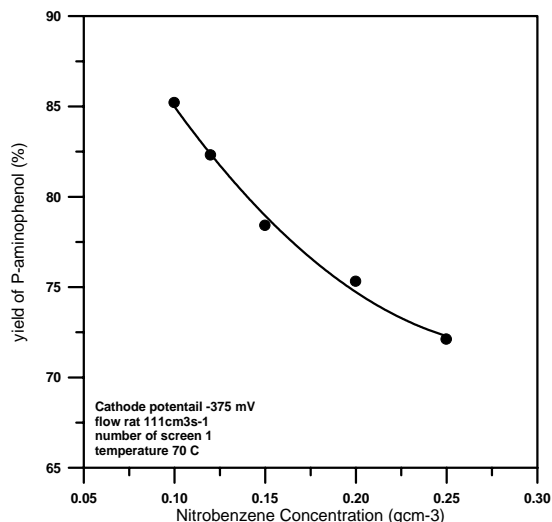


Fig. 10. The effect of nitrobenzene concentration on the yield of p-aminophenol

Table 2. Comparison between the potentiostatic operation at no. of screens=5 and 8

Parameters	No. of screens=5	No. of screens=8
Cell Current (A.)	20.5	27.5
Cell Voltage (V)	11.5	15.2
Yield (%)	80.0	73.0

Table 3. Comparison between galvanostatic and potentiostatic operation

Time(min.)	360	480	600	480 at constant electrode potential(-375mV)
Current (A)	20.00	20.00	20.00	20.50(initial)
Voltage (V)	12.00-11.60	12.00-11.30	12.00-10.80	11.50 (initial)
Yield (%)	73.00	72.80	70.70	80.00

3.4. Separation of the final product

A run was achieved under potentiostatic operation conditions for 30 hours. Samples of the catholyte or electrolysis solution were taken to determine the

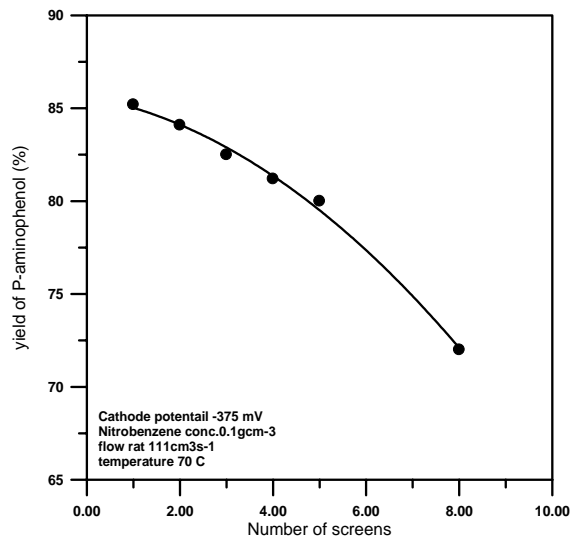


Fig. 11. The effect of screens number on the yield of p-aminophenol

3.3. Galvanostatic operation

Three production runs were achieved at initial cathode potential (-375mVvs.SCE), flow rate 111 cm³s⁻¹, nitrobenzene concentration 0.1gcm³, and screens number(5). The results are shown in table 3.

These results indicate that yield of p-aminophenol decreases with increasing time of electrolysis under constant current operation. The yield could be increased in constant current operation by increasing the temperature to 90°C where completely conversion of nitrobenzene to p-aminophenol could be obtained as mention in the previous work[17]. But due to the high evaporation rate of catholyte solution at this temperature we have to operate within 70-75°C. The potentiostatic operation gives higher yield than galvanostatic operation, therefore it would be useful to operate the system at controlled potential.

yield of p-aminophenol. the catholyte (14.5L) obtained from the electrolytic reduction of nitrobenzene were distilled at 92°C to remove the isopropanol (6.5L) then neutralized at temperature of 60-65°C, to

pH 4.5 with calcium carbonate. The calcium sulfate (1.5Kg) which formed was filtered off, the filtrate (7.5L) was extracted twice with (750cm³) of toluene. The aqua's phase was treated with (400g) of activated carbon and the latter filtered off. The activated carbon was regenerated by treating with 10% sodium hydroxide solution followed by 10% sulfuric acid solution at the boiling point of these solutions. To the filtrate obtained, (10g) of sodium hydrosulfite was added to prevent the oxidation of p-aminophenol followed by (400g) of acetic anhydride and (800g) sodium acetate at 40°C. The reaction mixture formed was cooled to 10°C with stirring and held at this temperature for 60 minutes, a crystalline precipitate (600g) of paracetamol was obtained after the filtration. The product was dried under vacuum at 80°C for 2 hours. The yield of p-aminophenol was 80% and the maximum conversion of p-aminophenol to paracetamol was 83%.

4. CONCLUSIONS

The rate and products of the electrolytic reduction of nitrobenzene depend mainly on the cathode potential. By improving the conditions of the operation like flow rate, nitrobenzene concentration and the number of screens this basic dependence is improved but not fundamentally altered. The polarization results indicate that the reaction is ECE type and diffusion controlled not mass control. The potentiostatic operation results gave a well defined effect of cathode potential, flow rate, nitrobenzene concentration and the total surface area of electrode on the yield of p-aminophenol, where the effect of cathode potential and flow rate are more effective than others. However, the best conditions for production of p-aminophenol under the potentiostatic operation were: Electrode potential (-375mV vs. SCE), Flow rate 111cm³ s⁻¹, Nitrobenzene concentration 0.1 gcm⁻³, and no. of screens 5 under this condition p-aminophenol has been achieved at yield 80%. The galvanostatic operation gives yield lower than potentiostatic operation therefore, it is recommended to operate the prototype system under controlled potential to utilize most of the fixed bed electrode area for the desired reaction by controlling the potential at the back of electrode within the value of nitrobenzene reduction potential.

Acknowledgment

I would like to express my sincere thanks to Arab Science and Technology Foundation represented by Dr. Abdullah Alnajjar for the financial support as a grant to achieve this project under the contract no.24-

06.A special thanks to Mr. Ammar M. Munir, the manager of grants departments, ASTF for his supporting this work which had been achieved under severe conditions of Iraq.

Nomenclature

A _a	actual surface area	cm ²
A _c	cross section area	cm ²
D	diameter of screen wire	cm
L	thickness of screen	cm
N	number of screens	----
NB. conc.	Nitrobenzene concentration	gcm ⁻³
PVDF	Polyvinyl dine fluoride	----
Q	flow rate of catholyte	cm ³ s ⁻¹
r	surface area to volume ratio of wire	cm ⁻¹
Re	Reylond number	----
S	specific surface area	cm ⁻¹
E	void fraction	----
ω	average weight area density of screen	gcm ⁻²
ρ _{cu}	density of copper	gcm ⁻³
ν	solution kinematics viscosity	cm ² .s
Φ	diameter of graphite disc	cm

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