*Korean J. Chem. Eng.*, **31**(5), 834-840 (2014)
DOI: 10.1007/s11814-013-0291-5

pISSN: 0256-1115
eISSN: 1975-7220

# Improved phenol adsorption from aqueous solution using electrically conducting adsorbents

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(*Received 19 September 2013 • accepted 23 December 2013*)

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**Abstract**−The electrically conducting and partially porous graphite based adsorbent (called NyexTM 2000) was tested for its adsorption capacity and electrochemical regeneration ability for the removal of phenol from aqueous solution. Nyex$^{TM}$ 2000 was tested in comparison with Nyex$^{TM}$ 1000, which is currently being used for a number of industrial waste water treatment applications. Nyex$^{TM}$ 1000 exhibited small adsorption capacity of 0.1 mg g$^{-1}$ for phenol because of having small specific surface area of 1 m$^2$ g$^{-1}$. In contrast, Nyex$^{TM}$ 2000 with specific surface area of 17 m$^2$ g$^{-1}$ delivered an adsorption capacity of 0.8 mg g$^{-1}$, which was eight-fold higher than that of Nyex$^{TM}$ 1000. Nyex$^{TM}$ 2000 was successfully electrochemically regenerated by passing a current of 0.5 A, charge passed of 31 C g$^{-1}$ for a treatment time of 45 minutes. These electrochemical parameters were comparable to Nyex$^{TM}$ 1000 for which a current of 0.5 A, charge passed of 5 C g$^{-1}$ for a treatment time of 20 minutes were applied for complete oxidation of adsorbed phenol. The comparatively high charge density was found to be required for Nyex$^{TM}$ 2000, which is justified with its higher adsorption capacity. The FTIR results validated the mineralization of adsorbed phenol into $CO_2$ and $H_2O$ except the formation of few by-products, which were in traces when compared with the concentration of phenol removed from aqueous solution. The electrical energy as required for electrochemical oxidation of phenol adsorbed onto Nyex$^{TM}$ 1000 & 2000 was found to be 214 and 196 J mg$^{-1}$, respectively. The comparatively low energy requirement for electrochemical oxidation using Nyex$^{TM}$ 2000 is consistent with its higher bed electrical conductivity, which is twice that of Nyex$^{TM}$ 1000.

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Keywords: Phenol, Improved Adsorption, Bed Electrical Conductivity, Reduced Electrical Cost

## INTRODUCTION

Phenol is used as a precursor for many useful compounds. It is highly toxic compound which can affect aquatic life **[1]**. It is discharged through effluent streams in significant amounts (200-600 mg L$^{-1}$) and has many adverse effects on human health even in small concentrations **[2]**. The intake of water contaminated with phenol into the human body causes protein degeneration, tissue erosion, paralysis of the central nervous system and also damages the kidney, liver and pancreas [2]. The level of toxicity of phenol was reviewed and found to be associated with hydrophobicity of free radicals [3].

The removal of phenol from water has been extensively investigated. Activated carbon is a well known adsorbent for the removal of organic pollutants from water. However, it has some limitations from environmental and economic perspective when executed for regeneration in order to recover its adsorption capacity [4]. Thermal regeneration may cause economic and environmental restrictions, and dumping is never been considered an environmentally friendly option [5]. Therefore, extensive research was carried out on alternatives such as chemical or solvent regeneration [6], electrochemical regeneration [7-12] or development of synthetic adsorbent materials [13]. Several commercial and synthetic polymeric materials were studied (acrylonitrile, 1,3 butadiene, styrene, PVC and poly (methyl methacrylate) to evaluate their ability to remove phenol from an aqueous solution. The results showed that styrene and 1,3 butadiene provided the highest adsorption capacity of 2.5 and 3 mg g$^{-1}$ for phenol removal [13]. The activated carbon was employed for the adsorptive removal of phenol and o-cresol from aqueous solutions. Adsorption capacities of 74 and 64 mg g$^{-1}$ were reported for phenol and o-cresol, respectively. The Freundlich, Langmuir and Redlich-Peterson models were tested to simulate the equilibrium isotherm data [14]. A study was carried out to investigate the liquid phase adsorption of phenol from water using silica gel, activated alumina, activated carbon, filtrasorb 400 and HiSiv 1000 [15]. Although activated carbon has remarkably higher specific surface area, it could not be characterized amongst electrically conducting adsorbent materials and, therefore, exhibited a significantly higher drop in voltage leading towards longer regeneration times when executed for adsorption followed by electrochemical regeneration studies [12].

To eliminate comparatively higher thermal regeneration cost of

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activated carbon coupled with material losses (5-10% on thermal regeneration), Brown et al. developed an electrically conducting bisulfate intercalated graphite material (reported as Nyex$^{TM}$ 100) and tested for a number of adsorption-electrochemical regeneration studies [8-11]. Nyex$^{TM}$ 100 showed significantly lower specific surface area (0.75 $m^2 g^{-1}$) that resulted small adsorption capacities for various organic pollutants [8-11]. The small adsorption capacity of Nyex$^{TM}$ 100 was considered to be compensated with its fast electrochemical regeneration obtained in minutes compared with activated carbon. Nyex$^{TM}$ 2000 showed a significant increase in adsorption capacity for acid violet 17 and humic acid. It also exhibited the ability to restore the same adsorption capacity through electrochemical regeneration [16,17]. The adsorption of phenol from aqueous solution onto carbon black was studied and it was found that the amount of adsorbed phenol increased with a decrease in number of oxygen compounds on the carbon surface [18].

The adsorptive removal of phenol from aqueous solution using partially porous graphite material (as developed and called Nyex 2000) has not been reported till date. The aim of this study is to compare the adsorption performance (Kinetics, isotherms and regeneration parameters) of Nyex$^{TM}$ 1000 & 2000 when employed for the removal of phenol from aqueous solution. In addition, surface investigations revealing the role of surface functional groups towards phenol adsorption have been carried out.

## MATERIALS AND METHODS

### 1. Materials

The standard solution of phenol was prepared in the laboratory using de-ionized water. Phenol of 99.5% was supplied by Sigma Aldrich, U.K. Adsorbent materials, called, Nyex$^{TM}$ 1000 & 2000 were supplied by the Arvia Technology Ltd. U.K. These were used without further treatment. The characterization of both adsorbent materials consisting of Boehm surface titration revealing surface functional groups, SEM for surface morphology, laser particle size analysis for average particle size measurement, bulk density, bed electrical conductivity and BET specific surface area have already been reported in [17].

### 2. Phenol Analysis

The quantitative analysis of phenol was conducted by high performance liquid chromatography (HPLC, Macclesfield, UK) by comparing the retention time of the standard solution of phenol. A Gemini C6 phenyl column, supplied by Phenomenex was used. The mobile phase was 30% methanol of analytical grade and 70% ultrapure water at a flow rate of 0.75 ml $min^{-1}$ through the column. The column temperature was maintained at 25 °C. A UV detector was used at a wavelength of 210 μm. All aqueous phenol samples were filtered through a 0.45 μm filter before their injection into the column.

### 3. Adsorption Kinetics and Isotherms

Adsorption kinetics and isotherm data were obtained by mixing a fixed mass of adsorbent with known concentration of phenol solution prepared in de-ionized water. The mixing was provided using a magnetic stirrer at 700 rpm in 1 L beaker for kinetic study and 250 ml flask for isotherm experiments. After adsorption the samples were collected for isotherm study, whereas for generating kinetic data, 5 ml samples were collected at regular time intervals and analyzed using HPLC after vacuum filtration using (VWR 413 grade).

### 4. Electrochemical Regeneration

Electrochemical regeneration of known mass of adsorbent loaded with phenol was carried out using a sequential batch electrochemical cell (Fig. 1). It was divided into anodic and cathodic compartments by a micro-porous Daramic 350 membrane (supplied by the Arvia Technology Ltd. UK). The cathode was made of perforated, 316 stainless steel and anode was a graphite plate. Each run of regeneration was started by filling a fresh aqueous solution of catholyte

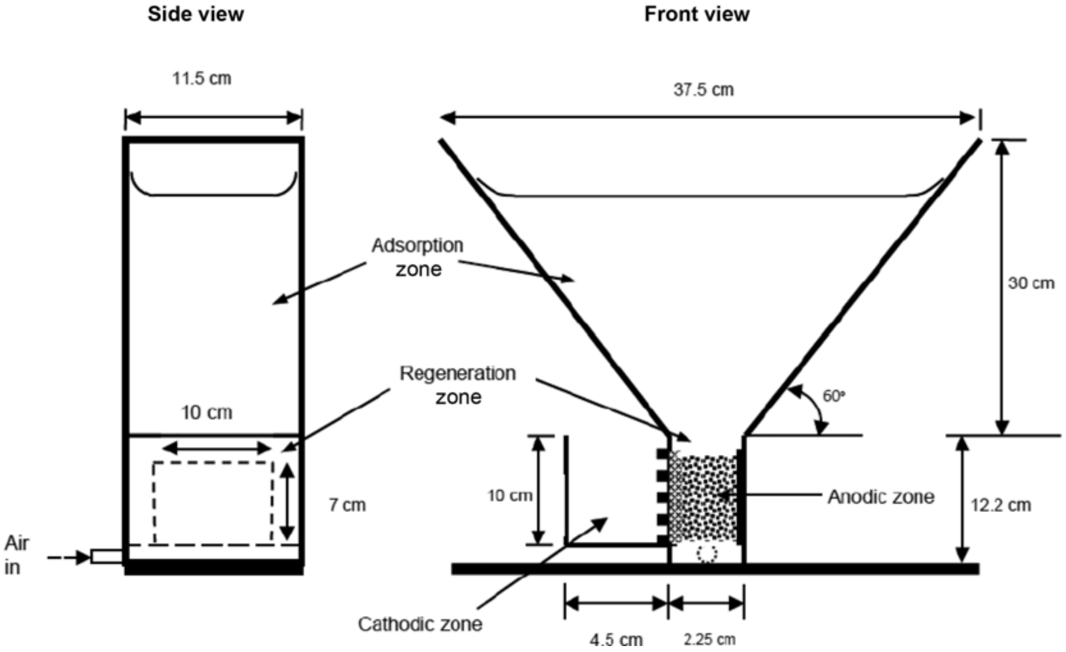

**Fig. 1. Schematic diagram of sequential batch electrochemical cell used adsorption followed by electrochemical regeneration for a no adsorption-regeneration cycles [16,17].**

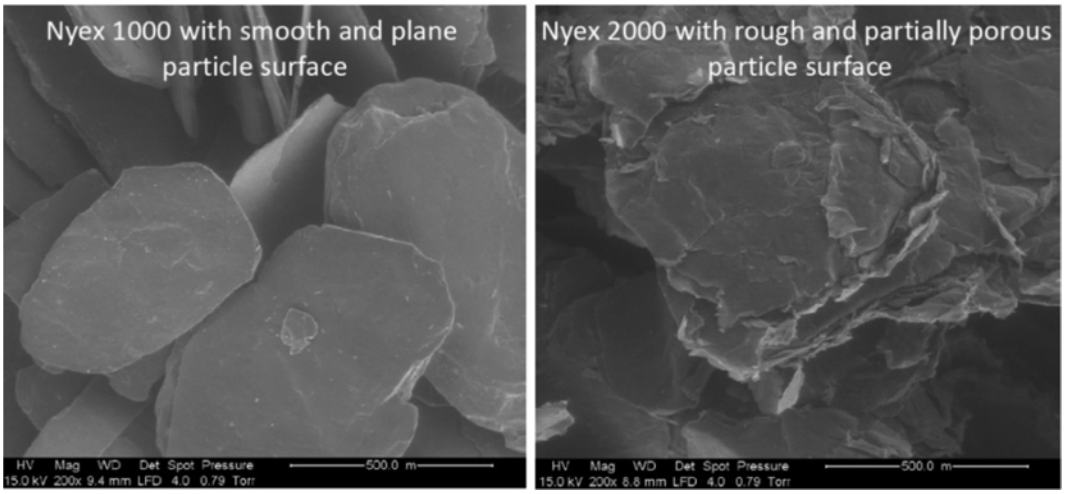

**Fig. 2. SEM images at 200x magnifications for Nyex$^{TM}$ 1000 & 2000 adsorbents.**

containing 0.3% w/w NaCl, whose pH was adjusted at 2 using HCl [16]. The procedure to determine the regeneration efficiency of both adsorbent materials when loaded with phenol was as follows;

i. Initial adsorption: A known mass of adsorbent material was added to 1 L phenol solution of known concentration. The mixture was agitated for 30 min by sparging with air. After adsorption, the air flow was stopped and allowed to settle into the anodic compartment of the cell. A sample of phenol solution was collected after adsorption and then analyzed for obtaining initial phenol loading ($q_i$) onto the adsorbent material.

ii. Electrochemical regeneration: When the adsorbent had settled in to the anodic zone, a DC current was supplied to the cell. After regeneration, the treated phenol solution was drained off.

iii. Re-adsorption: Regenerated adsorbent without further treatment was mixed by air sparging with fresh phenol solution of same concentration and volume as used in (i). After adsorption, the equilibrium phenol concentration and thus regenerated loading ($q_r$) were determined.

From the initial and regenerated adsorbent loadings, the regeneration efficiency was obtained as:

$$R=q_r/q_i * 100 \tag{1}$$

For multiple adsorption regeneration cycles, steps ii and iii were repeated.

## RESULTS AND DISCUSSIONS

### 1. Adsorbent Characterization

The investigations revealing characteristics of both the adsorbent materials have already been reported in [16]; however, for the sake of quick reference, these results are summarized here. Nyex$^{TM}$ 2000 was found to have almost double average particle size (784 μm) to that of Nyex$^{TM}$ 1000 (484 μm). The BET specific surface area for Nyex$^{TM}$ 2000 was found to be 17 $m^2 g^{-1}$ and it was improved by a factor of 16 folds when compared with 1 $m^2 g^{-1}$ for Nyex$^{TM}$ 1000. The significantly higher BET specific surface area and almost double average particle size of Nyex$^{TM}$ 2000 made it suitable adsorbent material for adsorption followed by electrochemical regeneration process. The increase in specific surface area of Nyex$^{TM}$ 2000 is associated with its rough surface morphology as evident in SEM image (Fig. 2).

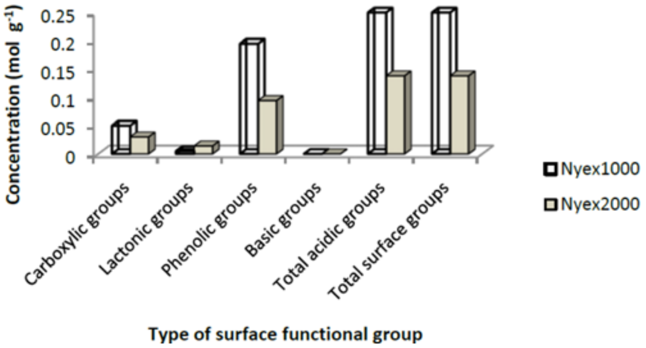

**Fig. 3. Concentration of surface functional groups as determined by Boehm surface titration [16].**

Fig. 3 presents a significant change in surface chemistry in terms of surface functional groups for both adsorbent materials, which has already been reported in [16]. The new surface chemistry was emerged as a result of chemical, thermal and physical steps underwent for the development of Nyex$^{TM}$ 2000 as originated from Nyex$^{TM}$ 1000. Nyex$^{TM}$ 2000 was found to have enriched with lactonic groups and about half the amount of phenolic groups when compared with Nyex$^{TM}$ 1000. The concentration of total acidic functional groups was found to be decreased from 0.25 mol $g^{-1}$ (Nyex$^{TM}$ 1000) to 0.138 mol $g^{-1}$ (Nyex$^{TM}$ 2000). However, still the particle surface of both the adsorbent materials was found to be dominating with acidic functional groups with no detectable basic functionality [16]. The proportion of phenolic groups was reported to be dominant at around 78 and 68% of the total surface functionality of Nyex$^{TM}$ 1000 & 2000 adsorbents, respectively.

The surface elemental analysis was conducted using X-ray (EDS) technique which showed C being a major constituent of Nyex$^{TM}$ 1000 with considerably higher concentration of O and S when compared with Nyex$^{TM}$ 2000. Traces of Mg, Al, Si and P were also detected as evident in Fig. 4. It is important to note that thermal processing of Nyex$^{TM}$ 1000 at 900 °C significantly altered the surface chemistry and caused to increase the proportion of C from 78 to 95% of

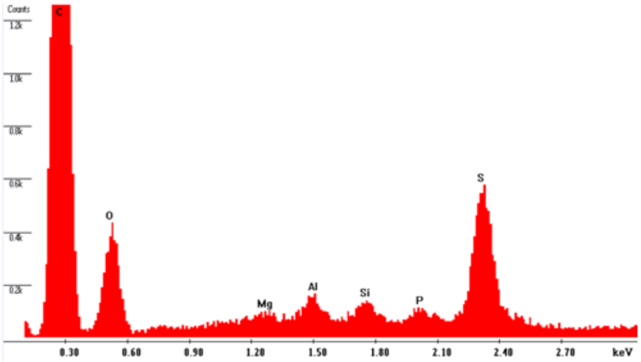

**Fig. 4. X-ray (EDS) spectrum for elemental analysis of Nyex$^{\text{TM}}$ 1000 adsorbent.**

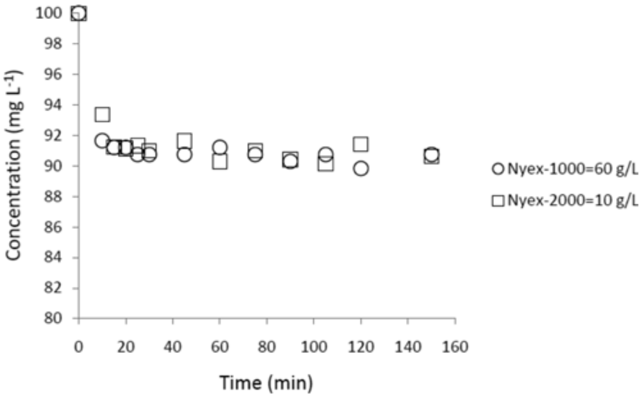

**Fig. 5. Adsorption kinetics for the adsorption of phenol from aqueous solution using Nyex$^{\text{TM}}$ 1000 & 2000 adsorbents using initial phenol concentration of 100 mg L$^{-1}$.**

Nyex$^{\text{TM}}$ 2000. The rest of the components were eliminated in the form of oxides as a result of thermal processing at such a high temperature.

The bed electrical conductivity of Nyex$^{\text{TM}}$ 2000 was found to be 1.6 S cm$^{-1}$ which is twice compared with 0.8 S cm$^{-1}$ for Nyex$^{\text{TM}}$ 1000. The higher bed electrical conductivity predicts reduced energy requirements for electrochemical regeneration [16].

## 2. Adsorption Kinetics & Isotherms

Kinetic study revealed an approximate time required to achieve equilibrium conditions for the removal of phenol onto both adsorbent materials. Results are shown in Fig. 5. Adsorption was found to be fast enough with 90% of the equilibrium being achieved within 5 minutes for each adsorbent. A gradual decrease in phenol concentration was observed during next 15 minutes as evident in Fig. 5. Therefore, a mixing time to achieve equilibrium was observed to be around 20 minutes for each adsorbent material. However, in view of the experimental errors, a contact time of 30 to 40 min can be speculated for achieving equilibrium conditions. Initial phenol concentration for both adsorbents was maintained at 100 mg L$^{-1}$. Brown et al. reported a mixing time of 30 minutes for achieving 90% of the equilibrium using starting phenol concentration of 250 mg L$^{-1}$ using Nyex$^{\text{TM}}$ 100 adsorbent [11]. The same equilibrium time was reported by Hussain for the removal of phenol with an initial concentration of 100 mg L$^{-1}$ using Nyex$^{\text{TM}}$ 1000 adsorbent [19]. It is worth considering that Nyex$^{\text{TM}}$ 2000 with an average particle size of 784 µm showed almost the same kinetics that of Nyex$^{\text{TM}}$ 1000 with an average particle size of 484 µm.

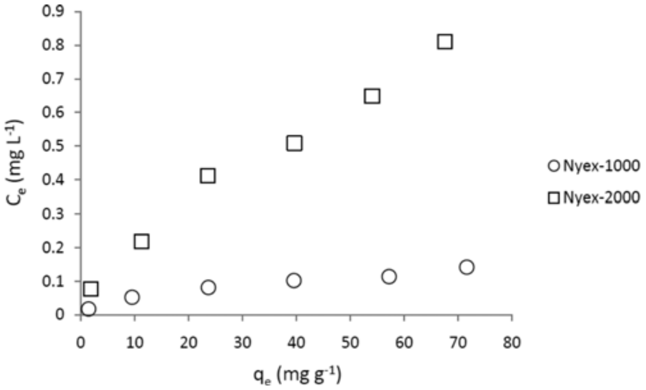

**Fig. 6. Adsorption isotherms for the adsorption of phenol onto Nyex$^{\text{TM}}$ 1000 & 2000 obtained using a contact time of 30 min to achieve equilibrium.**

Fig. 6 shows an isotherm data obtained for the adsorption of phenol onto both adsorbent materials using a mixing time of 30 min. It is evident in Fig. 6 that Nyex$^{\text{TM}}$ 2000 delivered an increased adsorptive capacity of 8 folds (0.8 mg g$^{-1}$) for phenol being an adsorbate when compared that of Nyex$^{\text{TM}}$ 1000 (0.1 mg g$^{-1}$). Thus, it can be concluded that Nyex$^{\text{TM}}$ 2000 is an effective adsorbent with comparatively large average particle size (784 µm) for the removal of phenol from its aqueous solution. This increase in adsorptive capacity can be explained with an increase in specific surface area of Nyex$^{\text{TM}}$ 2000 from 1 to 17 m$^2$ g$^{-1}$ which induced as a result of various chemical, thermal and mechanical treatments of Nyex$^{\text{TM}}$ 1000. In view of kinetic and isotherm data obtained for the removal of phenol using both the adsorbent materials, Nyex$^{\text{TM}}$ 2000 can be considered as a preferential adsorbent material due to its large average particle size and improved adsorption capacity. High bulk density and large average particle size can contribute to achieve rapid solid-liquid separation. The adsorption capacity of Nyex 2000 can be compared with Nyex 100, which also showed an adsorption capacity of 1 mg g$^{-1}$. Nyex 100 was characterized with an average particle size of 127 µm, which is significantly lower that of Nyex 2000 [11]. Brown et al. reported adsorption isotherms for mono, di and tri-chlorinated phenol compounds and observed that increasing the degree of chlorination strongly influenced the adsorption capacity of Nyex 100 [11]. A similar effect of improved adsorption of chlorinated phenols onto granular activated carbon has been reported by Streat et al [20,21]. Such a small particle size restricted its use in continuous adsorption-electrochemical regeneration process due to poor settling and it was, therefore, replaced with Nyex 1000 with an average particle size of 484 µm and adsorption capacity of 0.1 mg g$^{-1}$ for phenol. It is notable that adsorption capacity of Nyex materials is much lower to that of activated carbon as reported to be around 74 mg g$^{-1}$ by Nadia et al. [15].

Surface investigations of both adsorbent materials as evident in Figs. 3 and 4 revealed a significant difference in oxygen containing surface functional groups. The role of surface chemistry for the adsorption of phenol has prime importance as water molecules are expected to have electrostatic interaction with oxygen containing functional groups which were found to be dominant on the surface of Nyex$^{\text{TM}}$ 1000 compared with Nyex$^{\text{TM}}$ 2000 [18,22]. It is speculated due to polarity of hydrogen bonding in water molecules, which is considerably larger than phenol, and therefore strong polar molecules will induce affinity towards electrostatic interaction with surface functional groups of Nyex$^{TM}$ 1000 by suppressing the adsorption of phenol molecules [18,22]. The similar effect has already been reported in [18] for the adsorption of phenol onto the surface of carbon black used as adsorbent material. In view of the surface chemistry, Nyex$^{TM}$ 2000 is expected to be a more suitable material for the adsorption of phenol as it contained significantly lower concentration of oxygen containing surface functional groups as evident in Fig. 3. However, in addition to improved electrostatic interaction with phenol molecules, increased specific surface area of Nyex$^{TM}$ 2000, definitely, contributed as a leading factor to increase the adsorption capacity by a factor of eight-fold [22].

Adsorption isotherm data were analyzed using the Freundlich and Langmuir models. The Freundlich and Langmuir constants $K_F$, n, $q_m$ and $K_L$ indicating the adsorption capacity and intensity are summarized in Table 1. The Freundlich isotherm model was found to be in best-fit with higher values of coefficient of correlation, i.e., $R^2$=0.99. The Langmuir constant $q_m$ ensured the relatively low adsorption capacity of phenol onto both adsorbent materials [22].

**Table 1. The Freundlich and Langmuir constants for both Nyex$^{TM}$ adsorbents**

| Nyex$^{TM}$ 1000 & phenol | | | |
|---|---|---|---|
| Freundlich constants | | Langmuir constants | |
| n | 2 | $q_m$ (mg g$^{-1}$) | 0.1 |
| $K_f$ (L g$^{-1}$) | 63 | $K_L$ (L mg$^{-1}$) | 0.14 |
| Nyex$^{TM}$ 2000 & phenol | | | |
| Freundlich constants | | Langmuir constants | |
| n | 1.6 | $q_m$ (mg g$^{-1}$) | 0.71 |
| $K_f$ (L g$^{-1}$) | 19.9 | $K_L$ (L mg$^{-1}$) | 15.2 |

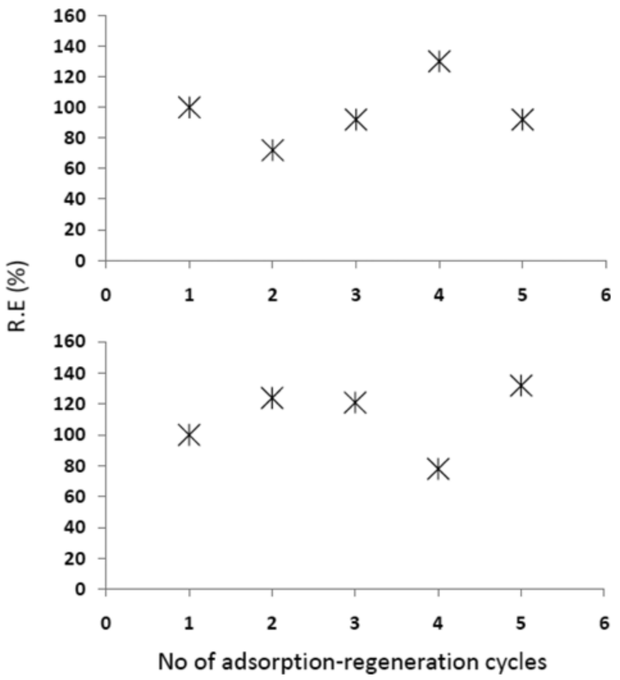

**Fig. 7. Electrochemical regeneration efficiency over five adsorption-regeneration cycles for the removal of phenol from aqueous solution as carried out in the electrochemical cell shown in Fig. 1**

### 3. Electrochemical Regeneration

Fig. 7 shows electrochemical regeneration efficiency obtained for a number of adsorption/regeneration cycles for both adsorbent materials. The regeneration efficiency remained consistent at around 100% for both adsorbents except first two cycles where a slight increase in adsorptive capacity was observed for Nyex$^{TM}$ 2000 unlike Nyex$^{TM}$ 1000, which was slightly lower than 100%. The increase in adsorption capacity during first two cycles for Nyex$^{TM}$ 2000 is consistent with the increase in adsorption capacity observed for the electrochemical regeneration of both Nyex$^{TM}$ 1000 and 2000 adsorbents when loaded with acid violet 17 [16] and humic acid [17]. The improved adsorption as a result of two adsorption-regeneration cycles can be attributed to lower pH (at around 2) existing in the anodic compartment of the electrochemical cell during regeneration process. The electrochemical parameters corresponding to an approximate 100% regeneration efficiency of Nyex$^{TM}$ 1000 and 2000 adsorbents were found to be 20 and 45 minutes being regeneration time at 7 and 10 mA cm$^{-2}$ current density and charge passed of 5 and 31 C g$^{-1}$, respectively. The actual charge passed of 5 and 31 C g$^{-1}$ for both adsorbents is comparable with theoretically calculated charge of 4 and 23 C g$^{-1}$ (using Eq. (2)), respectively.

$$C_6H_5OH+11H_2O \longrightarrow 6CO_2+28H^++28e^- \quad (2)$$

The charge passed can be compared to the charge of 36 C g$^{-1}$ with a current density of 10 mA cm$^{-2}$ as reported in the literature for the electrochemical regeneration of Nyex$^{TM}$ 100 with a similar adsorptive capacity to that of Nyex$^{TM}$ 1000 when loaded with phenol [11].

Comparatively less charge was found to be required for regenerating Nyex$^{TM}$ 1000 compared with Nyex$^{TM}$ 2000 as it had much lower loading of phenol. The electrical energy in accordance with charge consumed was found to be 214 and 196 J mg$^{-1}$ of adsorbed phenol onto Nyex$^{TM}$ 1000 and 2000 adsorbents, respectively, as shown in Fig. 8. The low energy consumption required for achieving 100% regeneration efficiency for Nyex$^{TM}$ 2000 is due to its higher bed- electrical conductivity of 0.16 S cm$^{-1}$ which is twice to that of Nyex$^{TM}$ 1000 at 0.8 S cm$^{-1}$ [16,17].

The FTIR spectrum as evident in Fig. 9 obtained for gas sample

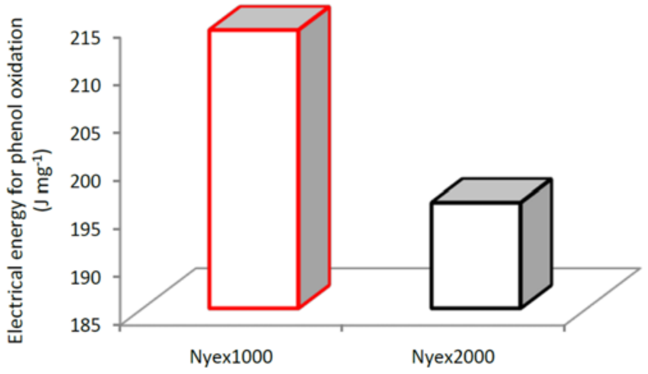

**Fig. 8. The comparison of electrical energy consumed for oxidizing 1 mg of phenol adsorbed on the surface of Nyex$^{TM}$ 1000 & 2000 adsorbents.**

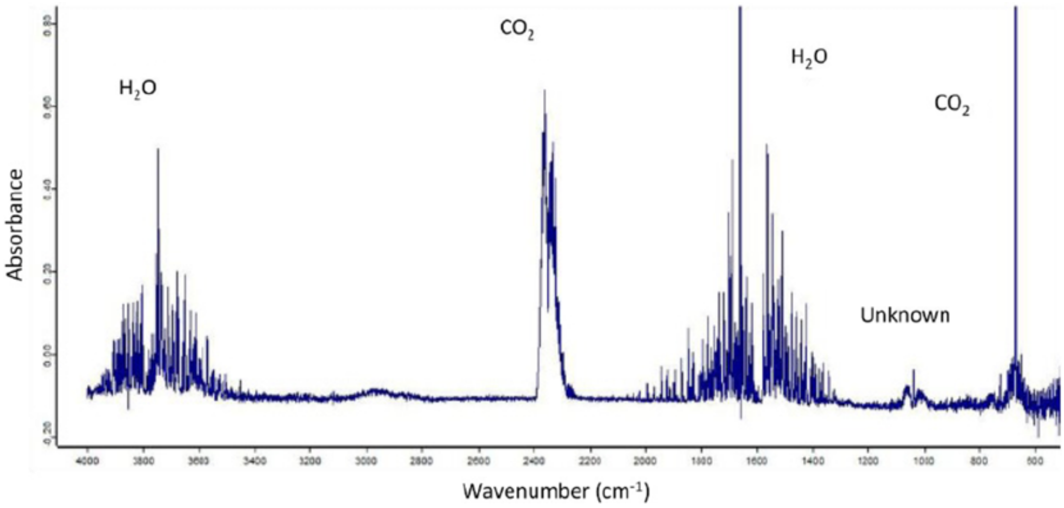

**Fig. 9. FTIR spectrum of gas collected during electrochemical regeneration of Nyex$^{TM}$ 1000 loaded with phenol [19].**

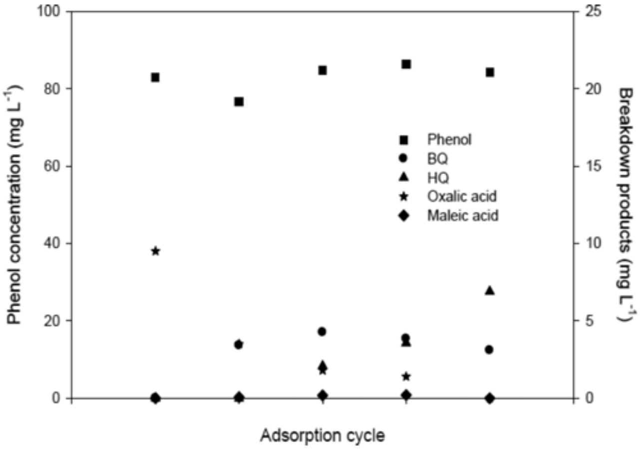

**Fig. 10. Concentration of electrochemical break down products of phenol in water after a serried of phenol adsorption cycles using Nyex$^{TM}$ 1000 at 10 mA cm$^{-2}$, regeneration time of 20 min with initial phenol concentration of 100 mg L$^{-1}$ [19-23].**

collected during the electrochemical regeneration of Nyex$^{TM}$ 1000 adsorbent material verified the formation of $CO_2$ and $H_2O$ as main oxidation products. However, further investigations dealing with oxidation products and by products as a result of electrochemical regeneration are in progress and will be reported in future as not being the scope of this paper [19].

To find the concentration of break-down products as were assumed to remain in the solution during electrochemical regeneration of Nyex$^{TM}$ 1000 saturated with phenol for which investigations were carried out.

As evident in Fig. 10, benzoquinone, hydroquinone, catechol, oxalic and maleic acids were identified as being intermediated oxidation products. These oxidation products were found in low concentrations compared to the concentration of phenol removed [19,23,24].

## CONCLUSIONS

Nyex$^{TM}$ 2000 was used as an adsorbent material for the removal of phenol from aqueous solution for which an increase in adsorption capacity of eight-fold was observed when compared with Nyex$^{TM}$ 1000. The increased surface area of Nyex 2000 (17 m$^2$ g$^{-1}$) was found to be a contributing factor for its improved adsorption capacity. The Freundlich isotherm model was found to be in best fit with experimental isotherm data. Nyex$^{TM}$ 2000 showed electrochemical regeneration efficiency at around 100% for which a current of 0.5 A and charge passed of 31 C g$^{-1}$ during a treatment time of 45 min were applied. Nyex$^{TM}$ 2000 consumed electrical energy of 196 J for electrochemical oxidation of adsorbed phenol which is comparable with 214 J as required for Nyex$^{TM}$ 1000. The lower electrical energy required for Nyex$^{TM}$ 2000 is attributed with its bed electrical conductivity, which is twice that of Nyex$^{TM}$ 1000. The electrochemical oxidation products were found to be $CO_2$ and $H_2O$ in gaseous phase with small concentration of benzoquinone, hydroquinone, oxalic and maleic acid as by products. However, the concentration of these by products was found to be much lower when compared with phenol removed from aqueous solution.

## ACKNOWLEDGEMENT

The work presented in this paper was sponsored by the Arvia Technology Ltd. UK. We are thankful to Arvia technology Ltd. UK., the University of Manchester, UK and University of the Punjab for extending their financial and logistic support for this project.

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