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## The effect of chemical activator and nanoscale pore-former on the performance of polyacrylonitrile-based carbon adsorbent for cationic dye removal from aqueous solution

Zahra Hosseini<sup>1</sup>, Morteza Nasiri<sup>1</sup>, Reza khoshbouy<sup>2\*</sup>

<sup>1</sup> Institute of Polymeric Materials and Faculty of Polymer Engineering, Sahand University of Technology, Tabriz, Iran

<sup>2</sup> Green Carbon Research Center, Faculty of Chemical Engineering, Sahand University of Technology, Tabriz, Iran

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### 1. ABSTRACT

This study investigated the effect of using polymethyl methacrylate (PMMA) as pore-forming agent along with potassium hydroxide (KOH) activator to synthesize polyacrylonitrile (PAN) carbon adsorbents with high porosity to enhance the adsorbent performance in methylene blue (MB) adsorption. PMMA nanoparticles were synthesized by mini-emulsion polymerization method. BET, FE-SEM-EDS and FTIR analyses were used to evaluate the carbon adsorbents. The specific surface area of PAN-based adsorbents increased from 10.6 to 393.6 m<sup>2</sup>/g with PMMA particles, and to 1897 m<sup>2</sup>/g after activation. FTIR results confirmed the removal of carbonyl groups and the presence of effective functional groups after activation. After simultaneous activation, the percentage of MB removal by the carbon adsorbent increased from 54% to 91%. The maximum adsorption capacity for the best sample was 2065 mg/g, which followed the Langmuir isotherm. The results showed that this combination method significantly increased the pore development, the adsorption capacity, and ultimately increased the performance of the carbon adsorbent.

**Keywords:** Polyacrylonitrile, Polymethyl Methacrylate, Potassium Hydroxide, Carbon Adsorbent, Methylene Blue.

### 2. INTRODUCTION

Methylene blue (MB) is a common cationic dye and a major pollutant in aquatic environments [1]. Porous carbon adsorbents have attracted significant attention for dye removal due to their high surface area and abundant adsorption sites [2]. Polyacrylonitrile (PAN) is considered a promising carbon precursor because of its high nitrogen content and excellent carbonization yield [3]. The incorporation of polymethyl methacrylate (PMMA) as a sacrificial template can promote pore formation [4], while KOH activation further enhances pore development and surface properties, resulting in highly porous carbon materials with superior adsorption performance [5].

### 3. MATERIALS AND METHODS

Acrylonitrile (AN) and ethylene glycol dimethacrylate (EGDMA) were purified using an activated alumina column to remove inhibitors. Methyl methacrylate (MMA) was washed twice with a 5 wt% sodium hydroxide solution followed by deionized water and subsequently purified by vacuum distillation. Potassium hydroxide (KOH), hydrochloric acid (HCl), dimethylformamide (DMF), and polyvinyl alcohol (PVA) were used as received without further purification.

#### 3.1. Synthesis and Activation of Porous Carbon Adsorbents

Polymethyl methacrylate (PMMA) nanoparticles were synthesized via a miniemulsion polymerization method and employed as a pore-forming agent. Subsequently, polyacrylonitrile (PAN) was polymerized in the presence of the PMMA

\* r.khoshbouy@sut.ac.ir

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nanoparticles to produce the initial PAN/PMMA composite structure. The resulting samples were dried, stabilized in air, and then carbonized at 700 °C under a nitrogen atmosphere. To enhance the specific surface area and develop the porous structure, chemical activation was carried out using KOH. Finally, the activated samples were washed with hydrochloric acid (HCl) and deionized water to remove residual potassium species, yielding a porous carbon adsorbent with a well-developed pore network.

## 4. RESULTS AND DISCUSSION

### 4.1. Analyses and characterizations

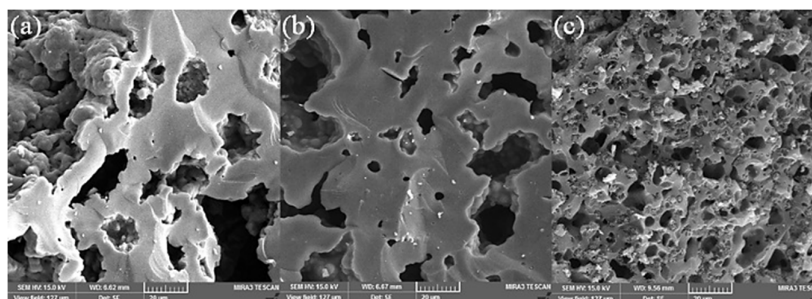
Table 1 summarizes the characteristics of the PAN-based carbon adsorbents synthesized under various preparation conditions. The sample nomenclature was assigned according to the precursor composition and processing parameters. For instance, PPM-C-700 refers to a sample synthesized from PAN in the presence of PMMA particles and subsequently carbonized (C) at 700 °C.

**Table 1.** Physicochemical characteristics of PAN-based carbon adsorbents synthesized under various preparation conditions

KOH: PAN	Activation temp (°C)	Carbonization tem (°C)	O (wt%)	C (wt%)	N (wt%)	V <sub>t</sub> (cm <sup>3</sup> /g)	V <sub>0</sub> (cm <sup>3</sup> /g)	S <sub>BET</sub> (m <sup>2</sup> /g)	Sample
-	-	700	6.4	73.6	19.9	0.004	0.003	10.6	PAN-C-700
-	-	700	6.3	66.8	26.8	0.21	0.13	393	PPM-C-700
1.5	700	-	15.8	71.5	12.3	0.93	0.4	1897	PPM-K-700

#### 4.1.1 FE-SEM Analyses

FE-SEM images (Fig. 1a) revealed that the carbonization of PAN led to pore formation due to the release of volatile gases, resulting in significant changes in surface morphology. The incorporation of PMMA particles into the PAN matrix (Fig. 1b) further increased the porosity through thermal decomposition and the evolution of volatile species, leading to the formation of larger and deeper pores and improving the permeability of the adsorbent. In the PPM-C-700 sample, pores with diameters ranging from approximately 2 - 5 μm were observed, confirming the effectiveness of PMMA as a pore-forming agent. Furthermore, the results demonstrated that controlling the PMMA content enabled the adjustment of pore size and volume, thereby enhancing the adsorption capacity. Following chemical activation with KOH (Fig. 1c), the porous structure became more developed, generating micro- and nanoscale pores with sizes ranging from approximately 120 nm - 2 μm. The presence of nanoscale pores within larger microscale pores indicates the formation of a hierarchical porous structure, which facilitates the penetration of the activating agent and consequently enhances the adsorption performance of the adsorbent.

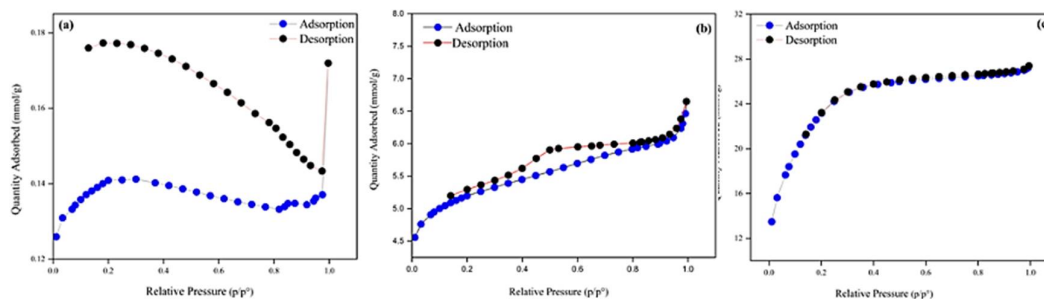


**Figure 1.** FE-SEM micrographs of the synthesized carbon adsorbents: (a) PAN-C-700, (b) PPM-C-700, and (c) PPM-K-700.

#### 4.1.2 BET Analyses

The PAN-C-700 sample (Fig. 2-a) exhibited a low specific surface area (10.64 m<sup>2</sup> g<sup>-1</sup>), low pore volume (0.003 cm<sup>3</sup> g<sup>-1</sup>), and a Type III adsorption isotherm with an H3 hysteresis loop, indicating limited porosity and weak adsorbate-adsorbent interactions. The incorporation of PMMA significantly enhanced the porous structure, increasing the specific surface area to 393 m<sup>2</sup> g<sup>-1</sup> and the pore volume to 0.003 cm<sup>3</sup> g<sup>-1</sup>, (Fig. 2-b). The resulting PPM-C-700 sample exhibited a hierarchical micro-mesoporous structure, characterized by a Type IV isotherm and an H4 hysteresis loop. This combination of

micropores and mesopores improved adsorption kinetics by providing both abundant adsorption sites and efficient mass-transfer pathways. Further chemical activation with KOH (Fig. 2-c) dramatically increased the specific surface area to 1897 m<sup>2</sup> g<sup>-1</sup> and the micropore volume to 0.4 cm<sup>3</sup> g<sup>-1</sup>, while maintaining a well-developed micro-mesoporous structure. The Type IV isotherm and H4 hysteresis loop of the activated PPM-K-700 sample confirmed the formation of an interconnected hierarchical porous network with enhanced adsorption potential.



**Figure 2.** Nitrogen adsorption–desorption isotherms of PAN-based carbon adsorbents: (a) PAN-C-700, (b) PPM-C-700, and (c) PPM-K-700.

#### 4.1.3 Adsorption performance of carbon adsorbents

The adsorption capacity of PAN-based carbon adsorbents toward MB was evaluated over 24 h. The PAN-C-700 sample exhibited a removal efficiency of 56.7%, which was attributed to its low pore volume. The incorporation of PMMA particles enhanced the porous structure and increased the adsorption capacity from 53.2 mg g<sup>-1</sup> (PAN-C-700) to 56.9 mg g<sup>-1</sup> (PPM-C-700) after 2 h. Further improvement was achieved through KOH activation, resulting in adsorption capacities of 86.5 mg g<sup>-1</sup> after 2 h and 91.4 mg g<sup>-1</sup> after 24 h for the PPM-K-700 sample. These results demonstrate that PMMA-induced porosity and KOH activation effectively enhance the adsorption performance of PAN-based carbon adsorbents for MB removal.

#### 4.1.4 Adsorption Isotherm Analysis

The adsorption behavior of MB on the PPM-K-700 adsorbent was evaluated using Langmuir, Freundlich, and Temkin isotherm models. The Langmuir model showed the best fit to the experimental data ( $R^2 > 0.995$ ), indicating monolayer adsorption on a homogeneous surface. Chemical activation not only enhanced the porosity of the carbon structure but also generated oxygen-containing functional groups on the surface. These functional groups provided additional active sites for electrostatic interactions and hydrogen bonding with positively charged MB molecules, leading to a significant improvement in adsorption capacity and dye removal efficiency.

### 5. CONCLUSION

The results demonstrated that PAN is an effective precursor for the preparation of porous carbon adsorbents. The incorporation of PMMA and KOH activation significantly improved porosity, specific surface area, and MB adsorption capacity. The Langmuir isotherm provided the best fit to the experimental data, indicating monolayer adsorption on a homogeneous surface. Owing to its well-developed porous structure and high adsorption performance, the synthesized carbon adsorbent shows strong potential for the removal of MB and other organic pollutants from aqueous solutions.

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