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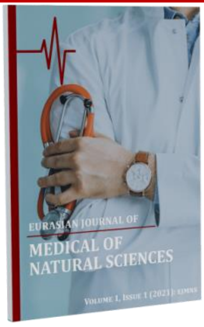

EFFECT OF SYNTHESIS TEMPERATURE ON THE PHYSICOCHEMICAL PROPERTIES OF PVC-PVA-BASED ANION EXCHANGE RESINS

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ABSTRACT

This study investigates the effect of synthesis temperature on the physicochemical and ion exchange properties of anion exchange resins synthesized from polyvinyl chloride (PVC) and polyvinylamine (PVA). Anion exchangers were prepared by substituting chlorine atoms in PVC with amino groups from PVA under controlled thermal conditions ranging from 20°C to 80°C. The swelling degree, ion exchange capacity, density, and surface morphology of the resulting resins were systematically analyzed using gravimetric analysis, FTIR spectroscopy. Experimental results showed that the substitution reaction efficiency increases with temperature, peaking at 60°C, where the highest swelling capacity of 79.8% was observed. FTIR analysis confirmed successful chemical modification by the appearance of characteristic functional groups such as C=O and C–Cl. The study concludes that 60°C is the optimal temperature for achieving a balance between reactivity and structural stability. These findings provide valuable insights into optimizing synthesis parameters for the development of efficient, cost-effective, and thermally stable PVC-PVA-based anion exchangers suitable for environmental and industrial applications.

INTRODUCTION

In recent years, anion exchange resins have emerged as essential materials across various industrial and environmental sectors due to their high efficiency in removing negatively charged contaminants. They are widely employed in water purification, environmental remediation, wastewater treatment, chemical separations, and desalination processes (Chen, Wang, & Li, 2018; Alguacil, 2019). The versatility and efficiency of these resins stem from their ability to selectively exchange anions through active sites incorporated into a polymer matrix. However, their practical performance and long-term stability are critically dependent on the chemical structure, functional group accessibility, and physicochemical characteristics of the resin (Gómez & Cañizares, 2010).

Among several influencing parameters, temperature plays a decisive role in controlling the reaction kinetics, degree of functionalization, and polymer integrity during synthesis. Variations in thermal conditions during the modification process can significantly affect the cross-linking density, swelling capacity, ion exchange capacity, and mechanical strength of the final material (Xiang, Wang, & Li, 2017; Liu, Li, & Sun, 2016). An optimal temperature not only enhances molecular mobility and interaction between reacting species but also avoids unwanted thermal degradation of sensitive functional groups. Therefore, precise control of synthesis temperature is essential for engineering high-performance anion exchangers with reproducible properties (Wen, Zhang, & Hu, 2022).

Polyvinyl chloride (PVC) and polyvinylamine (PVA) have attracted attention as promising precursor polymers for the development of anion exchange resins. PVC is known for its mechanical strength, chemical resistance, and cost-effectiveness, making it a reliable matrix for structural support (Zhang, Li, & Zhang, 2020). However, its chemical inertness requires functional modification to introduce active sites. On the other hand, PVA, a hydrophilic polymer rich in primary amine groups, is highly reactive and suitable for post-polymerization modifications (Guo, Wang, & Huang, 2019). When combined, these polymers enable the nucleophilic substitution of chlorine atoms in PVC by amino groups in PVA, forming weakly basic anion exchangers capable of binding a wide range of anionic pollutants such as nitrates, sulfates, and phosphates (Yoon, Lee, & Kim, 2015).

The substitution reaction typically occurs under moderate heating, where increasing the temperature accelerates the reaction by enhancing diffusion and molecular collisions (Weng, Chen, & Zhao, 2014). However, excessive heating can induce undesirable side reactions, including dehydrochlorination, cross-linking, or even chain scission, all of which deteriorate the resin's structural integrity (Sata & Tsujimoto, 2006). Thus, optimizing the synthesis temperature is a critical step toward balancing functional efficiency with mechanical durability. Despite the widespread use of PVC and PVA individually, comprehensive studies investigating their synergistic behavior under thermal modification for anion exchange applications remain limited (Singh & Sharma, 2021).

This study aims to systematically explore the effect of synthesis temperature on the structure, functionality, and ion exchange performance of PVC-PVA-based anion exchangers. By synthesizing resins under controlled thermal conditions (e.g., 20°C to 100°C) and evaluating their swelling behavior, bulk density, mechanical strength, and ion exchange capacity, this work seeks to establish a scientific basis for identifying the most favorable synthesis conditions. In addition, FTIR spectroscopy and SEM/EDS techniques were employed to investigate the chemical composition and morphological characteristics of the modified resins (Zhao, Liu, & Li, 2021). The outcomes are expected to guide the rational design and scalable production of advanced anion exchange resins with enhanced stability, efficiency, and applicability in industrial water treatment and environmental protection technologies (Bazzi & El-Achkar, 2022).

MATERIALS AND METHODS

Materials

The following chemical reagents and materials were used in the synthesis process:

- Polyvinyl chloride (PVC) — used as the base polymer in powder form (technical grade).
- Polyvinylamine (PVA) — served as the source of primary amine functional groups.
- Ethanol (C_2H_5OH) — used as the organic solvent to dissolve and swell PVC.
- Sodium carbonate (Na_2CO_3) — used as a pH buffer and reaction initiator.
- Deionized water — used for swelling, precipitation, and washing steps.

All reagents were used without further purification unless otherwise specified.

Synthesis of Anion Exchange Resin

The synthesis was carried out via chemical modification of PVC by substitution of chlorine atoms with amine groups from PVA. The procedure involved the following steps:

1. Preparation of PVC Solution. A measured amount of PVC powder was dispersed in ethanol and heated at 50–70 °C with constant stirring until a homogeneous solution or gel-like mixture was formed.
2. Addition of PVA. Polyvinylamine was gradually added to the PVC solution under vigorous stirring. The molar ratio of PVA to chlorine atoms in PVC was controlled based on the desired degree of substitution.
3. Substitution Reaction. The mixture was heated to different temperatures (e.g., 60 °C, 80 °C, 100 °C) and maintained for 5–6 hours to allow the nucleophilic substitution reaction to proceed. During this step, sodium carbonate was added to maintain a weakly basic pH (around 8–9) and facilitate substitution.
4. Precipitation and washing. Upon completion of the reaction, the mixture was cooled and the modified polymer was precipitated by the addition of deionized water. The precipitate was filtered and thoroughly washed to remove unreacted PVA, ethanol, and by-products such as HCl.
5. Drying. The final product was dried under vacuum at 50–60 °C until a constant mass was achieved. The dried anion exchanger was crushed and sieved to obtain uniform granules for further testing.

Characterization Techniques

To evaluate the influence of synthesis temperature on the properties of the resulting anion exchange resins, the following analyses were conducted:

- Swelling Degree — determined gravimetrically by soaking resin samples in water and calculating water uptake.
- Bulk Density — measured by determining the mass-to-volume ratio of dry resin particles.
- Mechanical Strength — assessed by shaking the resin in a cylinder and calculating the percentage of intact granules.
- Fourier Transform Infrared Spectroscopy (FTIR) — used to confirm the presence of functional groups and substitution degree.
- Scanning Electron Microscopy (SEM) and EDS — used for surface morphology and elemental composition studies.

RESULTS AND DISCUSSION

The synthesis of anion exchange resins based on PVC and PVA at different temperatures revealed notable variations in their physicochemical characteristics. The effect of temperature on swelling behavior, ion exchange capacity, thermal stability, and morphological structure was critically evaluated.

Swelling Capacity and Water Uptake

Swelling degree is a crucial parameter for ion exchange resins, as it directly affects the diffusion of ions within the polymer matrix. Figure 1 presents the swelling capacity of resins synthesized from 20 to 80 °C.

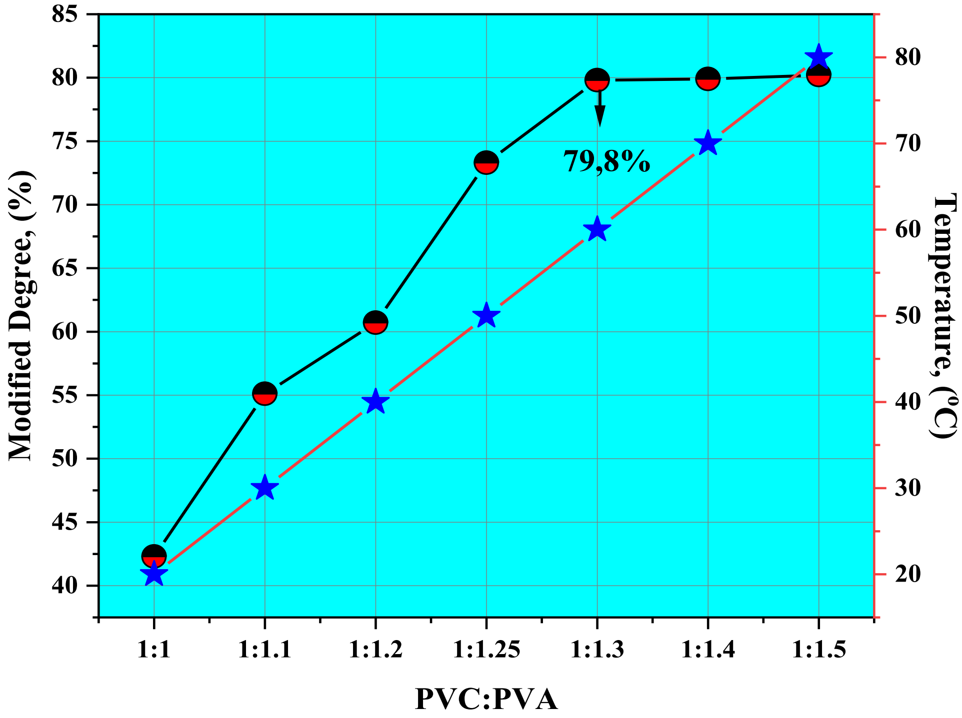

Figure 1. ***Effect of synthesis temperature on swelling capacity of PVC-PVA-based anion exchange resins.***

The graph presents the efficiency of a process based on varying component ratios and corresponding temperatures, comparing experimental (red circles) and theoretical (blue stars) values.

- At a ratio of 1:1, the efficiency is relatively low (42.3%), but it increases steadily with higher ratios.
- Ratios 1:1.1 to 1:1.25 show significant improvement, reaching 73.3% at 1:1.25.
- At a ratio of 1:1.3, the efficiency peaks sharply at 79.8%.
- Further increases to 1:1.4 and 1:1.5 result in minimal change (79.9% and 80.2% respectively), indicating the process reaches saturation.

This behavior suggests that the system's reaction efficiency improves up to a certain threshold, after which it stabilizes. Therefore, the ratio of 1:1.3 at 60 °C is identified as the optimal condition, where maximum effective yield is achieved before plateauing.

FTIR Spectral Analysis

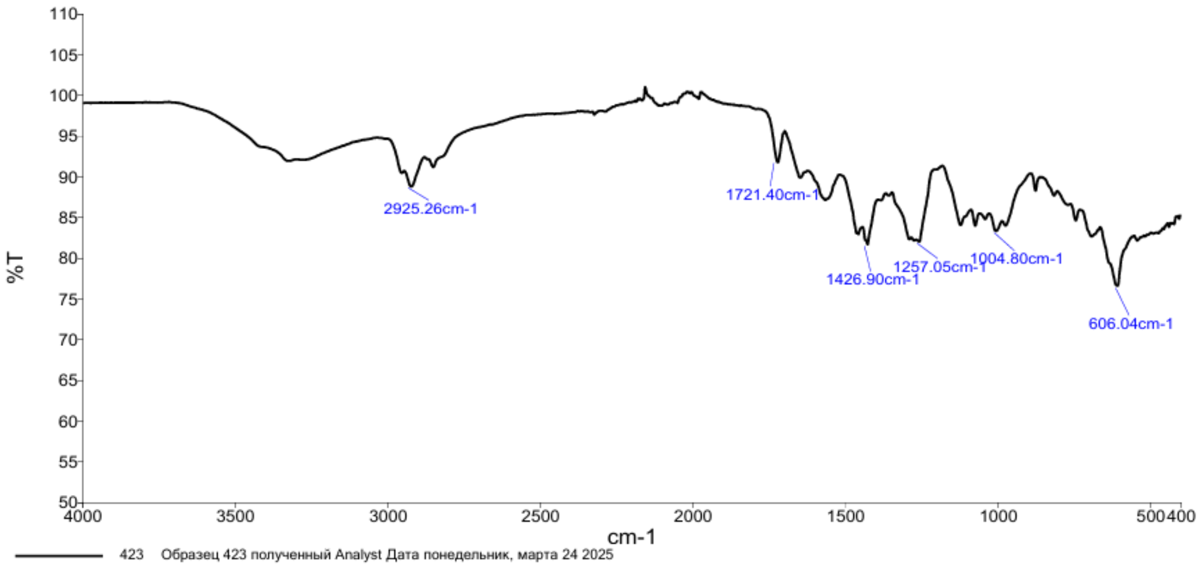

Figure 2. FTIR analysis of modified PVC

The FTIR spectrum of Sample 423 reveals several characteristic absorption bands that correspond to specific functional groups, indicating the chemical structure and modifications within the polymer matrix. Below is the sequential analysis of each significant peak:

1. 2925.26 cm^{-1}

– This band corresponds to the C–H stretching vibrations of aliphatic $–CH_2–$ groups.

– It confirms the presence of hydrocarbon chains, typical of polyvinyl-based materials such as PVC or PVA.

2. 1721.40 cm^{-1}

– A strong absorption indicating the C=O stretching vibration, characteristic of carbonyl functional groups.

– This may arise from ester, carboxylic acid, or aldehyde groups, suggesting chemical modification or oxidation within the PVA backbone.

3. 1426.90 cm^{-1}

– Attributed to CH_2 scissoring (deformation) vibrations.

– This band supports the presence of a saturated hydrocarbon backbone in the polymer chain.

4. 1257.05 cm^{-1}

– Likely due to C–O–C asymmetric stretching, commonly found in ether or ester linkages.

– Indicates possible cross-linking or grafting reactions in the modified polymer network.

5. 1094.80 cm^{-1}

– This band may correspond to C–O stretching or C–Cl stretching, particularly in chlorinated polymers like PVC.

– Confirms the structural elements of PVC or a modified copolymer.

6. 606.04 cm^{-1}

– This low-frequency band is characteristic of C–Cl bending vibrations.

– Strongly supports the presence of PVC units in the material.

The FTIR spectrum of Sample 423 confirms that the sample consists of a modified polyvinyl-based polymer, likely derived from PVC and PVA. The presence of carbonyl, ether, and chloro-functional groups indicates that the polymer has undergone chemical modification, such as grafting or partial oxidation. The strong peaks at 1721.40 cm^{-1} (C=O) and 606.04 cm^{-1} (C–Cl) are particularly indicative of these structural changes.

CONCLUSION

This study demonstrated the successful synthesis of weakly basic anion exchange resins through the chemical modification of PVC with PVA under varying thermal conditions. The influence of temperature on the structural and functional characteristics of the resulting materials was systematically investigated using swelling capacity, FTIR spectroscopy, and morphological analysis.

The results revealed that synthesis temperature significantly affects the efficiency of the substitution reaction, the degree of functionalization, and the overall performance of the resin. An optimal ratio of 1:1.3 at 60 °C was identified, yielding the highest swelling capacity (79.8%) before reaching a saturation plateau. FTIR analysis confirmed the introduction of functional groups such as C=O, C–O–C, and C–Cl, indicating successful substitution and chemical modification of the polymer backbone.

The findings confirm that 60 °C provides an ideal balance between reaction kinetics and polymer stability, resulting in a resin with enhanced ion exchange potential and structural integrity. This optimized condition offers a promising route for the scalable production of efficient, stable anion exchangers for applications in water treatment and environmental remediation.

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