

Development of Surfactant-Modified Natural Zeolite for Triclosan Adsorption

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Abstract: Triclosan (TCN) is a common antimicrobial and antifungal agent employed in pharmaceutical and personal care products (PPCPs). The persistent inability to completely remove TCN from wastewater has led to its accumulation across various wastewater matrices. Given the ecological and health consequences of TCN pollution, methods to prevent its entry into the wider aquatic environment are necessary. Due to its adsorption efficacy and abundance, Philippine natural zeolite (PNZ) is a promising candidate for environmental remediation as an effective adsorbent of charged contaminants. However, these natural zeolites have limited adsorption utility for non-polar species. To overcome this limitation, surfactant modification of natural zeolites was investigated to enable their adsorption of TCN. In this study, modification was achieved using cetylpyridinium chloride (CPyCl) and Arquad® 2HT-75 (ARQ) solutions prepared at 100% of the external cation exchange capacity of the zeolites, which yielded surfactant-modified natural zeolites (SMNZ) with a monolayer surfactant layer. Using Fourier-Transform Infrared Spectroscopy, Dynamic Light Scattering, Energy Dispersive X-ray Spectroscopy, and Simultaneous Thermal Analysis, surfactant modification was confirmed by the presence of distinct responses characteristic of surfactants in SMNZs that differentiate them from unmodified zeolite (UZ). TCN adsorption was investigated at varying pH (7, 8, and 9) and at varying adsorbent exposure times. Kinetic models (pseudo-first order, pseudo-second order, Elovich, and Weber-Morris interparticle diffusion) were fitted to the adsorption data, which revealed that TCN adsorption was best modeled by the pseudo-second order model. Surfactant modification yielded SMNZs with markedly improved TCN removal of up to 82% from a 50 ppm solution, in contrast to the unmodified zeolites, while reaching equilibration at >30 minutes. Overall, SMNZ showed effective removal of TCN and represents a potentially viable adsorbent for effective real-world wastewater treatment.

Keywords: Triclosan, personal care products, natural zeolite, wastewater, surfactant modification, adsorption.

1. Introduction

Triclosan (TCN) is a widely used antifungal and antibacterial compound in pharmaceutical and personal care products (PPCPs). The widespread use of these products has led to a notable presence of TCN in aquatic environments, where it is difficult to manage its harmful effects. Despite regulations limiting its concentrations in products, the compound remains recalcitrant to wastewater treatment. Removal via oxidation techniques yields more toxic and recalcitrant byproducts while achieving low mineralization levels (Luo et al., 2019; Z. Wang et al., 2024). Despite modern wastewater treatment methods, TCN concentrations in the parts-per-million range have been detected emanating from wastewater effluents and eventually entering biosolids and sediments, where they accumulate (Dar et al., 2022; Dhillon et al., 2015; Sun et al., 2023; Z. Wang et al., 2024). Its presence in vital aquatic matrices poses significant ecological and health concerns. TCN is classified as an endocrine-disrupting compound, with detrimental effects on hormonal homeostasis (Maksymowicz et al., 2021; Marques et al., 2022). Concerns have also been raised regarding its long half-life and its bioaccumulation potential (Y. Wang & Liang, 2021; Z. Wang et al., 2024).

Adsorption has been a mainstay technique and an effective means to prevent pollutants from entering aquatic matrices. It is widely emphasized that the use of underutilized, low-cost, and effective adsorbents is key to ensuring their sustainable use in wastewater treatment (Crini et al., 2019; Gupta et al., 2009; Ighalo et al., 2022; Younas et al., 2021). Among these, zeolites have established a niche as promising adsorbents with remarkable properties. Zeolites possess ion-exchange properties as well as adsorption properties conferred by their aluminosilicate structure (Pérez-Botella et al., 2022; Reeve & Fallowfield, 2018). As such, they have demonstrated their reliability as adsorbents of multiple key pollutants of concern (Cabrera et al., 2005; Zorpas et al., 2021). Most notably, Philippine natural zeolites (PNZs) have been shown to effectively remove heavy metals and a few organic pollutants from synthetic wastewater (Aguila & Ligaray, 2015; Faustino et al., 2024; Garcia et al., 2019; Gili et al., 2019; Olegario et al., 2019).

Despite these remarkable adsorption properties, raw zeolites weakly adsorb anionic and lipophilic contaminants (Apreutesei et al., 2008; Lu et al., 2022; Senila & Cadar, 2024). Among other techniques, surface modification of zeolites with cationic surfactants has made it possible to overcome this limitation with promising results. Cationic surfactants are molecules characterized by their long aliphatic tail terminated by a cationic head group (Reeve & Fallowfield, 2018). The positive charge conferred by its cationic head enables the modification of zeolite, which natively binds cations (Jiménez-Castañeda & Medina,

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2017). Depending on the amount of surfactant used, the zeolite surface acquires a non-polar surface provided by the tail of the surfactant or multiple cationic sites provided by the exposed head (De Gennaro, 2019; Smiljanić et al., 2020). This allows surfactant-modified natural zeolites (SMNZ) to remove recalcitrant dyes, pesticides, and drugs with varied physicochemical properties (Apreutesei et al., 2008; Nodehi et al., 2020; Shi et al., 2018; Smiljanić, Daković, et al., 2021). Previous studies have focused on the use of SMNZs to adsorb various pharmaceuticals, including non-steroidal anti-inflammatory drugs (NSAIDs), hormones, and antibiotics (Al-Mashaqbeh et al., 2021; Krajišnik et al., 2013; Smiljanić, Daković, et al., 2021). Among other surfactants, the use of cetylpyridinium chloride (CPyCl) and Arquad® 2HT-75 (ARQ) has been reported in clays and other minerals, where improved adsorption of various pollutants was observed (Liu et al., 2020; Phuekphong et al., 2021; Smiljanić et al., 2024; Zhang et al., 2019). Only a few studies have been reported that achieve modification of zeolites with these surfactants (Smiljanić, Daković, et al., 2021; Smiljanić et al., 2020).

2. Methodology

Materials and Reagents

Chemicals and Reagents. The natural zeolite was sourced from Pangasinan, Philippines, via LITHOS Manufacturing OPC Company. Triclosan (Tokyo Chemical Industry, T1872, CAS: 3380-34-5, MW = 289.54, >98.0% purity) was purchased from Theo-Pam Trading Corporation. The cationic surfactant Arquad® 2HT-75 (ARQ) [Sigma-Aldrich®, CAS: 61789-80-8, MW = 558.448] was purchased from Mega Source Scientific Supplies, and cetylpyridinium chloride (CPyCl) [Sigma-Aldrich®, CAS: 6004-24-6, MW = 358.00] from Krypton International Sales and Services Incorporated.

Zeolite Precursor Preparation

The zeolite samples were cleaned of debris in five rinses. For each rinse, the zeolite was submerged in deionized water and shaken at 180 rpm for 25 minutes using a DaiHan Scientific® Sho-1D Digital Orbital Shaker. The cleaned zeolites were retrieved by decanting the liquid, oven-dried at 105°C for 24 hours using a Memmert UN-160 lab oven, and then sieved with an analytical mesh to separate particles <125 µm to yield unmodified zeolite samples (UZ) (Tran et al., 2018).

Sodium-saturated zeolites (NaZ) were prepared as suitable precursors to generate SMNZs (Shi et al., 2018; Tran et al., 2018). NaZs were obtained by mixing 1.0 g of UZ with 100 mL of 0.3 M NaOH solution under continuous shaking at 150 rpm for 24 hours to establish a uniform cation presence on the surface. The crude NaZ was recovered by vacuum filtration through qualitative filter paper and transferred to ceramic crucibles before oven-drying at 105°C for 24 hours.

Surfactant Modification

External Cation Exchange Capacity (ECEC) Determination. The ECEC value for NaZs was determined as previously described in the literature (Cappelletti et al., 2017; De Gennaro et al., 2016). In

duplicate, a NaZ-surfactant mixture was prepared by combining 6.0 g NaZ (mass of zeolite in extractant, m_{NaZ}) in 50 mL (volume of the extract, V_{extract}) of 20 mM HDTMA-Br and shaken for 24 hours at 180 rpm. The extract was collected by centrifugation at 3000 rpm for 5 minutes and recovery of the supernatant into separate plastic containers. Following the methods described in the Standard Methods for Examination of Water and Wastewater (SMEWW 3120-B), the concentration of cations [(C_{cat}) : Na^+ , K^+ , Ca^{2+} , and Mg^{2+}] was measured in mg/L by Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES, Prodigy 7, Teledyne) (Baird & Bridgewater, 2017; Smiljanić et al., 2020). The ECEC value of the zeolite was then expressed as the sum of the cations (mEq) per unit mass of zeolite:

$$mEq_{\text{cat}} = \frac{C_{\text{cat}} \times V_{\text{extract}} \times \text{valence}_{\text{cat}}}{MW_{\text{cat}}} \quad (1)$$

$$ECEC \text{ per gram zeolite} = \frac{\sum mEq_{\text{cat}}}{m_{\text{NaZ}}} \quad (2)$$

where $\text{valence}_{\text{cat}}$ is the valence and MW_{cat} is the molecular weight of the respective cation.

Surfactant Modification of NaZ. Surfactant modification was conducted as described in the literature (Hailu et al., 2017; Shi et al., 2018; Smiljanić, Daković, et al., 2021; Tran et al., 2018). Modification was accomplished by mixing NaZs into the surfactant-enriched solutions of the selected surfactants: CPyCl and ARQ. The concentration of the surfactant solution for modification ($M_{\text{surfactant}}$) was calculated from this equation:

$$M_{\text{surfactant}} = \frac{ECEC \text{ per gram zeolite} \times m_{\text{NaZ},m}}{\text{valence}_{\text{surfactant}} \times (V_{\text{extract}})} \quad (3)$$

where m_{NaZ} is the mass of NaZ designated for modification and $\text{valence}_{\text{surfactant}}$ is the valence of the cationic surfactant. In this work, modification was achieved at a ratio of 4.0 g of NaZ: 100 mL of surfactant solution. Based on the previous ECEC calculation, the solution was prepared at exactly 100% of the ECEC value. To promote modification, the mixture was allowed to equilibrate with shaking at 180 rpm for 48 hours. The SMNZs were allowed to settle for 12 hours and separated by vacuum filtration through qualitative filter paper. The separated SMNZs were subjected to deionized water washes to eliminate any remaining surfactant, which was confirmed by the absence of frothing when the samples were shaken in water (Hailu et al., 2017). For 72 hours, the SMNZs were then allowed to air-dry at 25°C before storage in glass containers in a desiccator. The process yielded CPyCl modified zeolites (CMZ) and ARQ-modified zeolites (AMZ) with a monolayer surfactant surface.

Adsorption Studies

Standard TCN solutions (64, 32, 16, 8, 4, 2, and 1 ppm) were prepared from a stock solution dissolved in 20% ethanol.

Using UV-Vis spectrophotometry (BMG Labtech FLUOstar Omega), a spectral scan of the highest concentration standard (64 ppm) was performed from 220-500 nm to determine the absorbance maximum ($\lambda_{\max}$) of TCN. Blank correction was performed by simultaneous reading of the solvent alone.

All adsorption setups were prepared with an adsorbent dosage ($m_{\text{adsorbent}}$): 10.0 mg, synthetic wastewater volume (V_{drug}): 10 mL, and shaking: 150 rpm. Setups were prepared in 100 mL Erlenmeyer flasks. The effects of pH on TCN adsorption were investigated at pH levels of 7, 8, and 9 by allowing triplicate setups with a synthetic wastewater concentration (C_0) of 50 ppm TCN to equilibrate for 24 hours. Kinetic parameters of adsorption were evaluated by analyzing batch setups with a synthetic wastewater

concentration (C_0) of 40 ppm TCN in triplicate at varying contact times: 1, 5, 10, 20, 30, 60, 120, and 180 minutes. Three aliquots were withdrawn from each setup and analyzed for residual TCN concentrations at $\lambda_{\max, \text{TCN}} = 280$ nm. The amount of TCN sorbed onto the adsorbent (Q_t , in mg g^{-1}) and percent removal (%R) were calculated as follows:

$$Q_t = \frac{V_{\text{drug}}(C_0 - C_t)}{m_{\text{adsorbent}}} \quad (4)$$

$$\%R = \frac{(C_0 - C_t)}{C_0} \times 100 \quad (5)$$

where t = equilibration time; C_0 = initial TCN concentration; and C_t = TCN concentration at t . Adsorption data were fitted to the following adsorption kinetic models in Table 1:

Table 1. Adsorption kinetic models (Sahoo & Prelo, 2020; J. Wang & Guo, 2020; William Kajjumba et al., 2019)

Model	Equation	Variables
Pseudo-first order (PFO)	$\ln(Q_e - Q_t) = \ln Q_e - K_1 t$ (6) Plot: $\ln(Q_e - Q_t) = f(t)$	
Pseudo-second order (PSO)	$\frac{t}{Q_t} = \frac{1}{Q_e^2 K_2} + \frac{t}{Q_e}$ Plot: $\frac{t}{Q_t} = f(t)$	Q_t (mg/g) = amount of adsorbate per unit adsorbent mass at time t Q_e (mg/L) = adsorption capacity at equilibrium K_1 (1/min) = PFO rate constant
Elovich Kinetic Model	$Q_t = \frac{1}{\beta} \ln \ln(\alpha\beta) + \frac{1}{\beta} \ln \ln(t)$; Eq. 10 Plot: $Q_t = f[\ln \ln(t)]$	t (min) = adsorption time K_2 (g/mg*min) = PSO rate constant t (min) = adsorption time
Weber & Morris Interparticle Diffusion Model	$Q_t = k_i t^{0.5} + C_i$; Eq. 12 Plot: $Q_t = f(t^{0.5})$	

The coefficient of determination (R^2) was used to evaluate the goodness of fit of the linear models.

Characterization Techniques

Both UZ and SMNZs were subjected to various characterization techniques, as similarly conducted in the literature (Smiljanić, Daković, et al., 2021). Detection of pertinent functional groups was performed by Fourier transform infrared spectroscopy (FTIR) using a Shimadzu® IRSpirit™ equipped with a QATR™-S single-reflection ATR accessory. Zeta potential (ζ) was measured with a Horiba Scientific® nanoPartica SZ-100V2 nanoparticle analyzer to determine the charge state of the material's surface ($n_{\text{sample}} = 1.475$, $n_{\text{medium}} = 1.333$). Thermal analysis was conducted using a PerkinElmer® STA 6000 instrument. During analysis, the samples were held in ceramic crucibles and analyzed from 30 °C to 900 °C with a heating rate of 10.00 °C/min while nitrogen gas purged the compartment at 20 mL/min. To investigate the surface characteristics after modification, imaging by scanning electron microscopy (SEM) was performed with a Hitachi® SU-1510. The surface elemental composition was also determined using a ThermoFisher Scientific™ UltraDry Energy Dispersive X-ray (EDX) detector with an accelerating voltage of 15.0 kV. The samples were sputter-coated with platinum using a Hitachi® MC1000 ion sputter coater for 30 seconds before analysis. X-ray diffraction (XRD, Rigaku™ SmartLab SE diffractometer) was performed with

a Cu-K α radiation source ($\lambda = 1548$ nm) with the diffraction angle variation set to 0.4° per min over the 2θ range of 5° to 80°.

The assessment of the electrical state of the SMNZ at varying pH and ionic strengths was carried out using the pH drift method as described previously (Bajpai & Tyagi, 2010; J. Lin et al., 2011). Varying distilled water solutions were prepared over the pH range of 2-12 (2-unit interval) with minor adjustments made using 0.01 M HNO₃/KOH drops. Potassium nitrate (KNO₃) was also added to 0.1 M to serve as an inert background electrolyte. The initial pH (pH_i) was determined using the PASCO PS-3204 Wireless pH sensor. In 50-mL plastic containers, 20 mg of each adsorbent (UZ or SMNZ) was mixed in 10 mL of the prepared salt solutions and allowed to incubate for 24 hours under continuous shaking at 180 rpm. The supernatant was filtered through qualitative filter paper and analyzed for its final pH (pH_f). Analysis was conducted in triplicate over three trials of the procedure. The difference between pH_f and pH_i (pH drift, ΔpH) was plotted against its respective pH_i and the point of zero charge (pH_{pzc}) was determined as the pH_i value where $\Delta\text{pH} = 0$.

3. Results

Adsorbent Physicochemical Properties

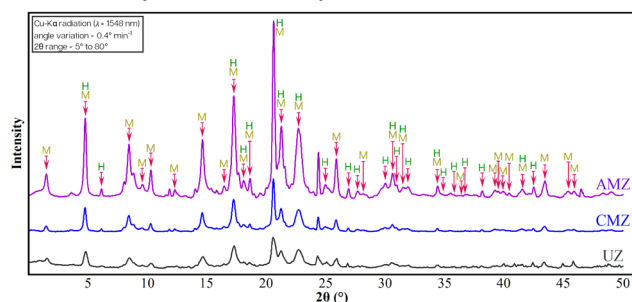


Figure 1. X-ray diffractograms of AMZ, CMZ, and UZ. Matching peaks for *heulandite* (H) and *mordenite* (M) are labeled accordingly.

Modification was evident from distinctive variations in the physicochemical properties observed between the UZs and the synthesized SMNZs. As indicated in Figure 1, XRD analysis revealed peaks characteristic of mordenite (PDF 00-060-0846) and heulandite (PDF 00-053-1176). Surfactant modification of UZs did not produce any apparent shifts in the peak positions, which

have been previously observed in surfactant modification of zeolites with other surfactants (Bagheri et al., 2024; Hailu et al., 2017). This is expected, as modification occurs only at the zeolite's surface without causing any changes in the zeolite TO₄ tetrahedral cage. As noted previously, modification occurs only by ion exchange of the positively charged surfactants onto the negatively charged zeolite surface (Jiménez-Castañeda & Medina, 2017). The large hydrophobic surfactant tails are unable to access the interior of the mineral because of the very small pore sizes.

SEM images of UZ, sodium-saturated zeolites NaZ, CMZ, and AMZ are presented in Figure 2. The image of UZ is consistent with the observation of crystalline mordenite particles and the apparently layered plate-like heulandite that constitutes the starting material (Korkuna et al., 2006; Panek et al., 2014; Sakizci & Özgül Tanriverdi, 2015). After surfactant modification, CMZ₁₀₀₀ and AMZ₁₀₀₀ micrographs display slight changes in surface characteristics and morphologies. Similar observations have also been reported in which surfactant modification appears to impart a smoother surface onto the zeolite, with its crystal edges appearing less sharp (Bagheri et al., 2024; Hailu et al., 2017).

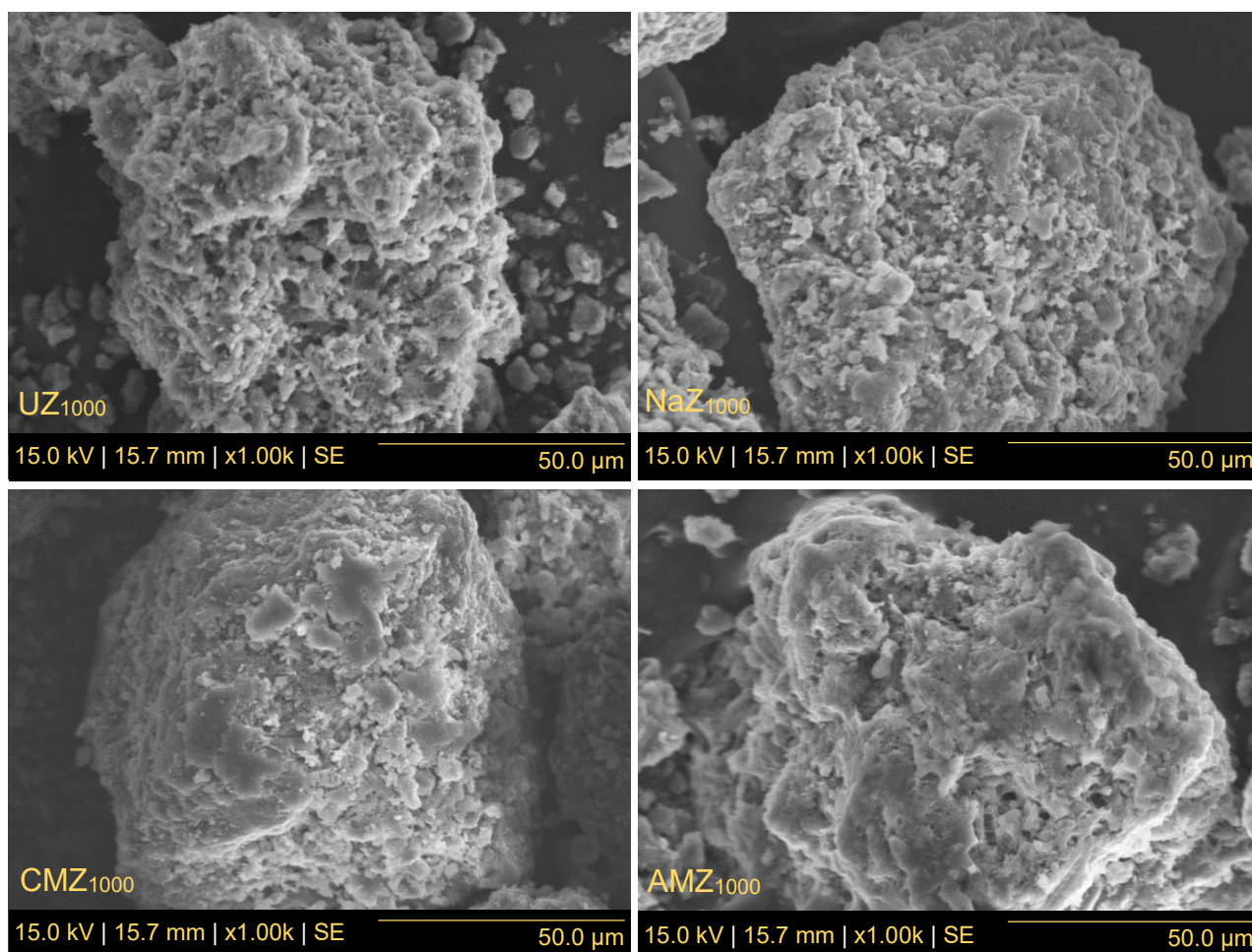


Figure 2. SEM micrographs of: UZ, NaZ, CMZ, and AMZ at 1000X magnification.

During sodium saturation, the sodium ions exchanged readily with cations natively present on the surface of the zeolite to improve the cation exchange during surfactant modification. As shown in Table 2, evidence of sodium saturation was observed in the elevated % Na levels in the NaZ samples compared with UZ.

After modification, there was also an apparent decrease in % Na, which is indicative of the cation exchange occurring with the surfactant molecules. Moreover, consistent with the surfactant modification is the increase in % C, which is attributable to the carbon-rich surfactant molecules (Nodehi et al., 2020).

Table 2. Surface elemental presence in UZ, NaZ, CMZ, and AMZ measured by EDX.

Element	Weight % $\pm$ SEM			
	UZ	NaZ	CMZ	AMZ
C	0.355 $\pm$ 0.225	1.650 $\pm$ 0.622	7.024 $\pm$ 1.033	5.695 $\pm$ 0.383
O	39.697 $\pm$ 0.840	43.335 $\pm$ 1.284	40.260 $\pm$ 0.951	42.042 $\pm$ 1.739
Na	1.416 $\pm$ 0.081	4.874 $\pm$ 1.145	1.305 $\pm$ 0.053	1.346 $\pm$ 0.217
Mg	0.396 $\pm$ 0.101	0.328 $\pm$ 0.110	0.547 $\pm$ 0.030	0.442 $\pm$ 0.086
Al	8.008 $\pm$ 0.571	5.305 $\pm$ 0.831	6.312 $\pm$ 0.182	6.099 $\pm$ 0.535
Si	38.776 $\pm$ 0.670	29.321 $\pm$ 5.781	33.417 $\pm$ 1.142	32.143 $\pm$ 3.418
Cl	0.024 $\pm$ 0.024	-	0.030 $\pm$ 0.030	0.050 $\pm$ 0.033
K	3.133 $\pm$ 0.982	0.697 $\pm$ 0.087	0.691 $\pm$ 0.054	0.672 $\pm$ 0.062
Ca	3.608 $\pm$ 1.205	10.107 $\pm$ 6.607	4.952 $\pm$ 0.747	6.601 $\pm$ 2.763
Ti	0.528 $\pm$ 0.209	0.517 $\pm$ 0.073	0.552 $\pm$ 0.176	0.586 $\pm$ 0.096
Fe	3.913 $\pm$ 0.285	3.867 $\pm$ 0.881	4.482 $\pm$ 0.461	4.324 $\pm$ 0.758
Ba	0.145 $\pm$ 0.145	-	0.369 $\pm$ 0.242	-

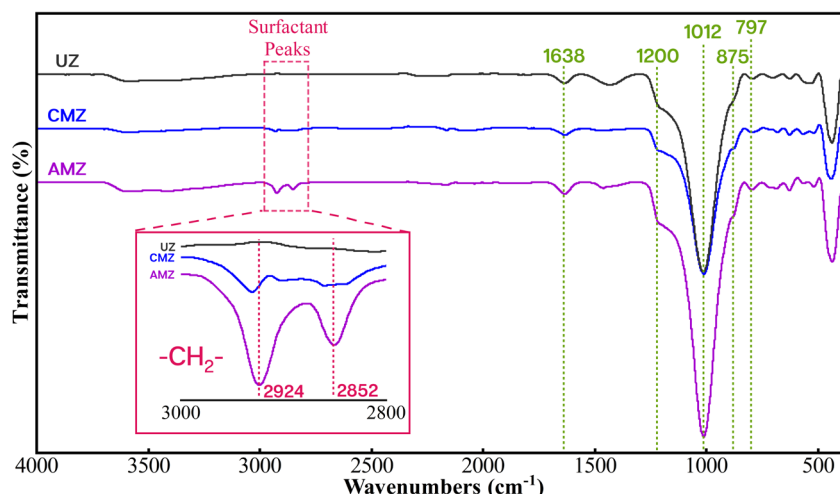


Figure 3. FTIR transmittance spectra of UZ, CMZ, and AMZ. Indicated by the green dotted lines are the wavenumbers characteristic of zeolites.

Significant functional groups are evident in the FTIR-ATR spectra of UZs, CMZs, and AMZs, which are given in Figure 3. Evidently, surfactant modification has left most of the zeolite structure unperturbed, signified by the absence of shifts in band placement among the samples analyzed. The TO_4 ($\text{T} = \text{Al}$ or Si) tetrahedra of zeolite are evidenced in all the samples: a distinct peak at 1012 cm^{-1} with a shoulder at 1200 cm^{-1} (T-O asymmetric stretching), a band at 875 cm^{-1} (T-O-T bending), and pseudolattice vibrations at 700-500 cm^{-1} (Korkuna et al., 2006; Smiljanić, Daković, et al., 2021). A trace amount of water is also present, given by a weak band at 1638 cm^{-1} (deformation vibration of absorbed water) and

a weak broad band over the 3400-3600 cm^{-1} range (OH vibration of water), which may be contributed mainly by zeolite O-H stretching. By contrast, surfactant peaks are given by the SMNZs, indicated by two small peaks at 2852 cm^{-1} and 2924 cm^{-1} . As reported in previous literature that has achieved modification with the same surfactants, these peaks are given by symmetric and asymmetric C-H stretching, which are characteristic of both CPyCl and ARQ acyl tails (Korkuna et al., 2006; Smiljanić et al., 2020; Smiljanić, Daković, et al., 2021). The peaks are more pronounced in the latter, which may be largely due to the two-tailed nature of ARQ as opposed to the single-tailed CPyCl.

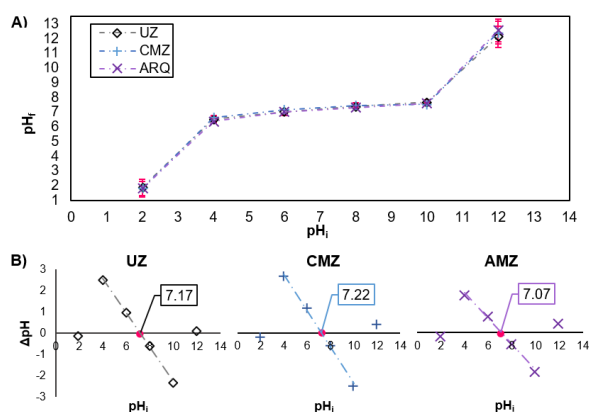


Figure 4. (A) Plots of $pH_f = f(pH_i)$ and (B) $\Delta pH = f(pH_i)$ of: UZ ($\diamond$), CMZ ($\square$), and AMZ ($\times$). Points of zero charge are indicated in (B) by the red point ($\bullet$).

The charged state assumed by an adsorbent's surface at varying pH can determine its affinity toward various charged species. Over the tested pH range of 2 to 12, it is evident that modification did not affect the pH_{pzc} of UZ. As shown in Figure 4.A., both the base material UZ and SMNZs exhibit a similar $pH_f = f(pH_i)$ plot with an apparent buffering region from pH 4 to pH 10. Further establishing this point is the lack of significant deviation between the pH_{pzc} obtained in $\Delta pH = f(pH_i)$ plots shown in Figure 4.B. Composites similar to the synthesized SMNZs (Smiljanić, Daković, et al., 2021; Smiljanić, De Gennaro, et al., 2021; Smiljanić et al., 2020) and other surfactants (Bagheri et al., 2024; Li, 1999; J. Lin et al., 2011) also displayed similar behavior. As indicated in the previously cited literature, the results show that SMNZs maintain similar acid and base properties as the starting UZs over the tested pH range.

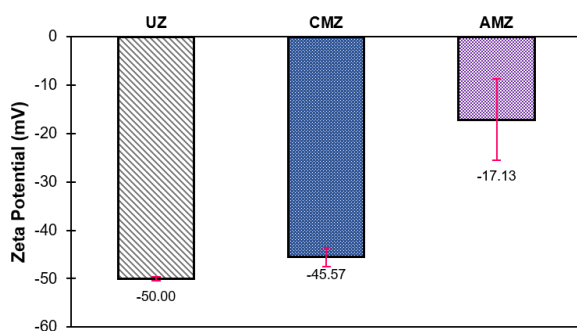


Figure 5. Zeta potentials of UZ, AMZ, and CMZ. Indices of refraction used in measurement: $n_{\text{sample}} = 1.475$, $n_{\text{medium}} = 1.333$.

Zeta potential measurement assesses the charge carried by the material by virtue of its surface functional groups. As depicted in Figure 5, UZ exhibited a negative charge, which likely arises from the residual negative charge of the zeolite surface (Armbruster & Gunter, 2001; Korkuna et al., 2006; Margeta et al., 2013). SMNZs displayed an increasingly positive charge with ARQ modification, achieving a more nearly complete monolayer morphology by virtue of its proximity to zero. As observed in previous literature, CMZ did not show the same degree of shift, likely due to the adsorption of micellar CPyCl, thereby leaving some zeolite surface uncovered (Smiljanić, Daković, et al., 2021).

Assessment of the thermal properties of the zeolite base materials and SMNZs allows the determination of thermal events, which would further confirm surfactant modification. The DTA profiles of UZ, NaZ, and SMNZs are shown in Figure 6.A., all of which depict characteristic zeolite thermal events: an endothermic peak between 30–125 °C, which is attributable to water molecules occluded in the zeolite undergoing evaporation (De Gennaro et al., 2016; Hailu et al., 2017) and an exothermic peak at 600–900 °C where zeolite ablation occurs, followed by its recrystallization into another phase (Afzal, 2000; Król & Jeleń, 2021; Smiljanić et al., 2020). Aside from these peaks, an additional unique exothermic peak is evident in the SMNZs between 220 and 420 °C, which has been identified by previous studies as the region where degradation of the organic surfactants occurs (Cappelletti et al., 2017; De Gennaro et al., 2016; Smiljanić et al., 2020). These peaks coincide with weight loss events observed in the TG-DTG profiles provided in Figure 6.B. Overall, residual weights of the analyzed samples ranged from 81% to 83.29%. Observable in all materials are two weight loss events characteristic of the zeolite base material occurring concurrently with the dehydration and zeolite structure ablation, events that are also reported in the literature (De Gennaro et al., 2016; Hailu et al., 2017). By contrast, both SMNZs showed an additional weight loss of 2% occurring within 308–342 °C, which coincides with the organic degradation peaks observed. This confirms the thermal stability of the synthesized SMNZs, which highlights their potential for use in water treatment.

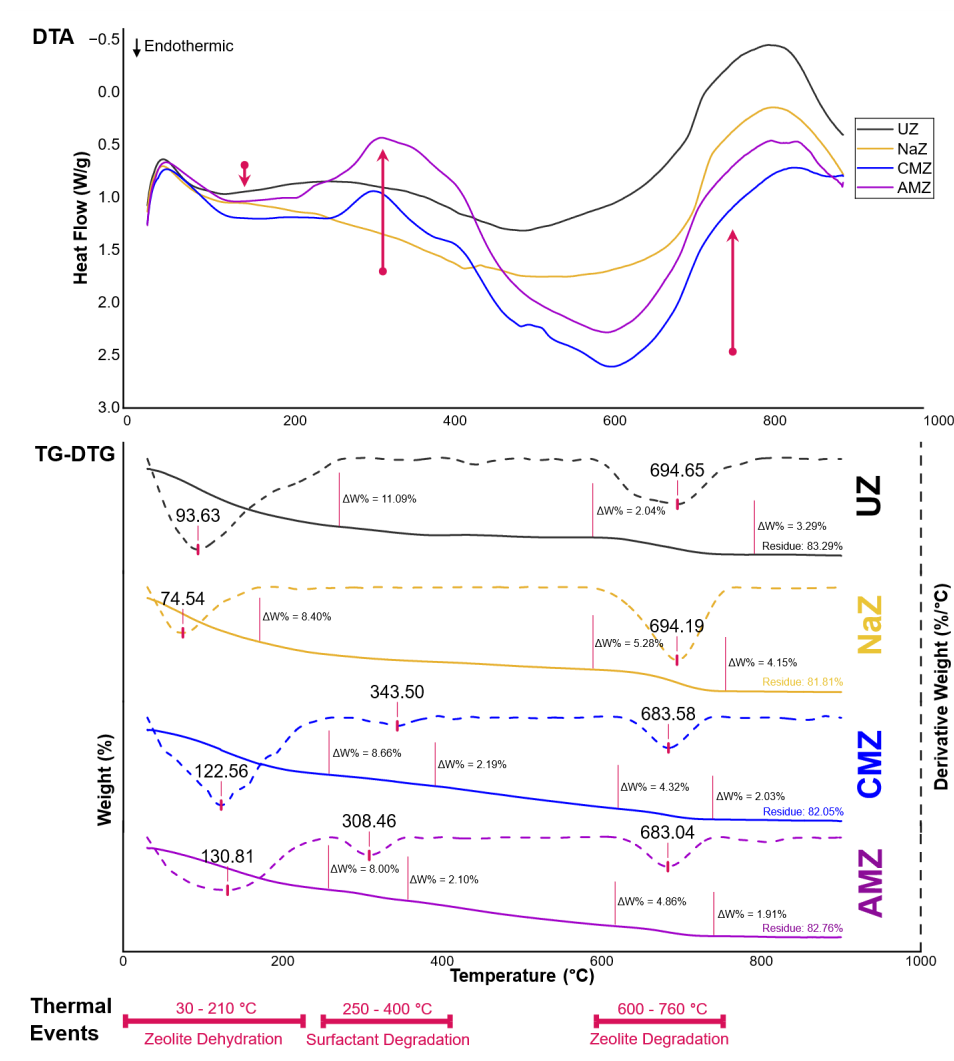


Figure 6. Simultaneous thermal analysis (STA) of UZ (-), NaZ (-), CMZ (-), and AMZ (-): (A) DTA and (B) TG (indicated by a solid line) and DTG (indicated by dashed lines); temperatures of thermal events are indicated accordingly. The following parameters were used in the analysis: purged with N₂ gas (20 mL/min) and analyzed from 30 to 900 °C.

Adsorption Studies

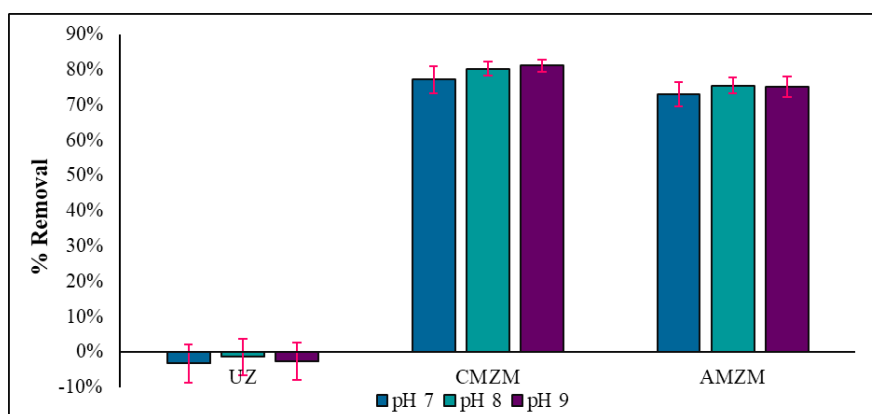


Figure 7. TCN % removal by UZ, AMZ, and CMZ at varying pH. The standard errors of the mean are minimal (< 5.0% for CMZM and AMZM, and below 6.0% for UZ).

Figure 7 illustrates the percent removal of TCN by AMZ and CMZ compared to UZ at the given pH range. The results show that both modified zeolites had significantly improved adsorption efficacy with a percentage removal of up to 81.22% for CMZ ($Q_t = 20.30$ mg/g) and 75.76% for AMZ ($Q_t = 18.91$ mg/g) as opposed to UZ, with close to 0% removal. This adsorption performance is comparable to that of activated carbon (AC) from waste biomass at 80.77% (Mohd Khori et al., 2018). Moreover, the adsorption performance of SMNZ is notable, as it was achieved with a higher pollutant concentration of 50 ppm compared to only 5 ppm for AC, and with a lower dose of adsorbent at 1 mg/mL versus 2 mg/mL for AC. Overall, it is apparent that the SMNZs may adsorb more TCN while requiring less of the material, which indicates its potential as a sustainable and effective adsorbent. With regard to the TCN removal of UZ, the negative values are close to zero,

owing to the negligible adsorption of TCN on UZ. While CPyCl (one-tailed) and ARQ (two-tailed) had varying surfactant morphologies, there was no evident difference in their adsorption capacities as validated by a t-test.

The adsorption of certain species may be a pH-dependent process, as this may alter the charge of the adsorbate, thereby affecting its affinity to the adsorbent (Peñafiel et al., 2024). With a pK_a of 7.9, triclosan assumes a negative charge at pH above 7.9 and is neutral at pH below the pK_a . In this study, TCN adsorption at pH 7, 8, and 9 was investigated. In the investigated pH range, no significant effect on TCN adsorption was observed, as confirmed by ANOVA. Consequently, a subsequent study of the adsorption kinetics of the prepared SMNZs was conducted at pH 7.0 for practicality.

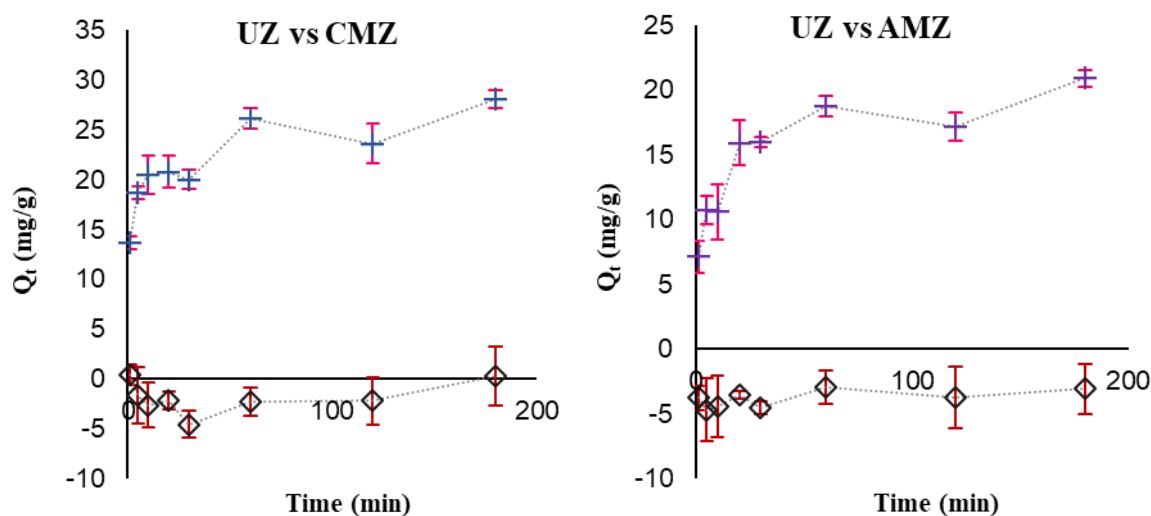


Figure 8. Adsorption of TCN per mass adsorbent (Q_t) by CMZ and AMZ compared to UZ.

The variation in the amount of TCN adsorbed across different exposure times is given in Figure 8, which consistently shows an improved adsorption of TCN. The cumulative increase in the surface area of the SMNZ and its non-polar character have likely produced this observation (Apreutesei et al., 2008; Cappelletti et al., 2017; Smiljanić, De Gennaro, et al., 2021). Due to the organic character of TCN, it tends to partition into non-polar substrates such as organics, as described in previous literature (Agyin-

Birikorang et al., 2010; Chen et al., 2022; Wilson et al., 2009). Linear fitting of the adsorption data onto adsorption kinetic isotherms was conducted to extract information on the possible TCN adsorption mechanism. Each SMNZ appears to achieve equilibration at 60 minutes with a small further increase in adsorption capacity beyond this time. Each fitting model is visualized in relation to the adsorption data in Figures 9 and 10, while the parameters for each model are given in Tables 3 and 4.

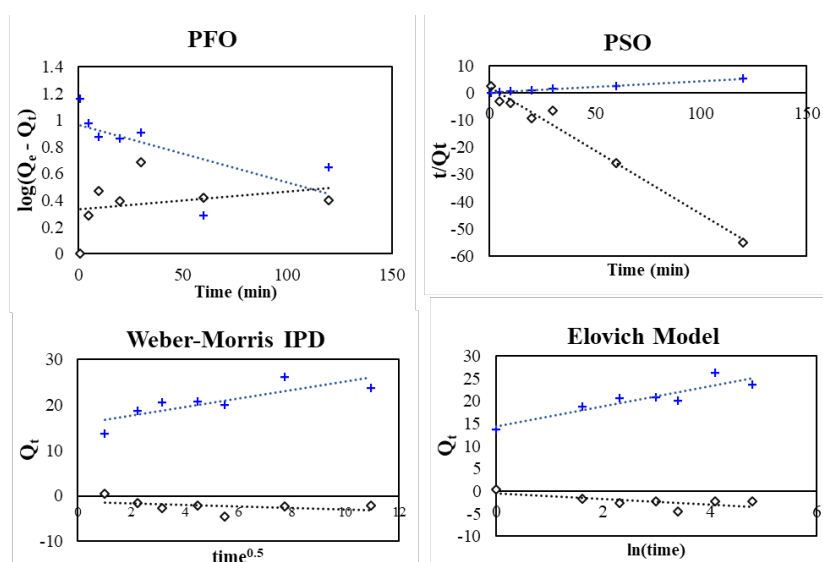


Figure 9. Kinetic isotherm fitting of TCN adsorption by CMZ vs. UZ.

Table 3. Kinetic parameters of TCN adsorption of UZ vs CMZ via Linear Curve Fitting.

Model	Parameter	UZ	CMZ
Pseudo-First Order (PFO) Model	R^2	0.0737	0.4328
	$K_1 (\text{min}^{-1})$	-0.013	0.0043
	$Q_e (\text{mg g}^{-1})$	1.3949	2.6327
Pseudo-Second Order (PSO) Model	R^2	0.9817	0.9941
	$K_2 (\text{g mg}^{-1} \text{min}^{-1})$	0.1112	0.02127
	$Q_e (\text{mg/g})$	-2.1445	24.3309
Elovich Kinetic Model	R^2	0.4327	0.8387
	$^{\dagger}\alpha (\text{mg g}^{-1} \text{min}^{-1})$	-1.4177	1421.1170
	$^{\ddagger}\beta (\text{g mg}^{-1})$	-1.6767	0.4487
Weber-Morris IPD Model	R^2	0.1759	0.6743
	$C_i (\text{mg g}^{-1})$	-1.2587	15.812
	$^tK_i (\text{mg g}^{-1} \text{min}^{0.5})$	-0.1783	0.9371

$^{\dagger}\alpha$ = Initial adsorption rate

$^{\ddagger}\beta$ = Initial desorption rate

tK_i = Diffusion Rate constant

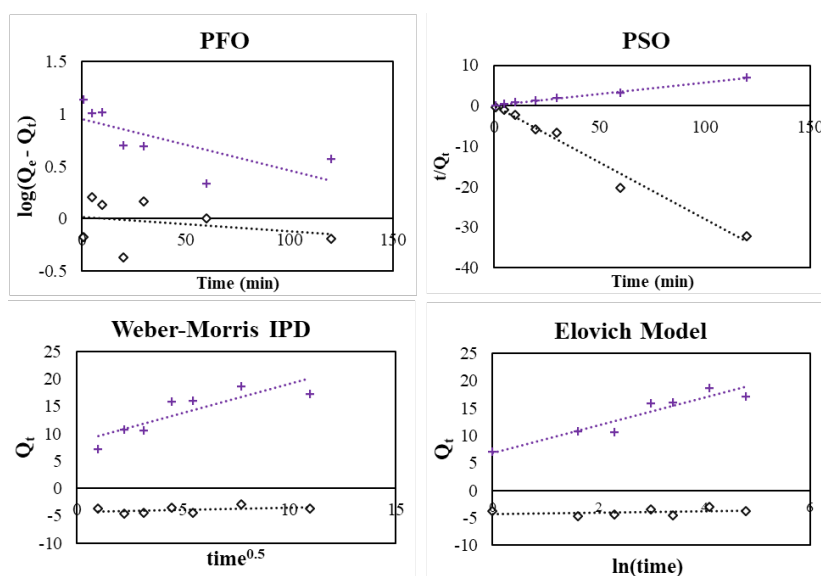


Figure 10. Kinetic isotherm fitting of TCN adsorption by AMZ vs. UZ.

Table 4. Kinetic parameters of TCN adsorption of UZ vs AMZ via Linear Curve Fitting.

Model	Parameter	UZ	AMZ
Pseudo-First Order (PFO) Model	R^2	0.0712	0.5166
	K_1 (min^{-1})	0.0014	0.0049
	Q_e (mg g^{-1})	1.0147	2.5831
Pseudo-Second Order (PSO) Model	R^2	0.9798	0.9951
	K_2 ($\text{g mg}^{-1} \text{min}^{-1}$)	1.0630	0.01863
	Q_e (mg/g)	-3.5701	17.9533
Elovich Kinetic Model	R^2	0.1076	0.8872
	$^{\dagger}\alpha$ ($\text{mg g}^{-1} \text{min}^{-1}$)	4.1423×10^{-16}	39.3174
	$^{\ddagger}\beta$ (g mg^{-1})	7.7700	0.3997
Weber-Morris IPD Model	R^2	0.1878	0.7254
	C_i (mg g^{-1})	-4.341	8.4382
	tK_i ($\text{mg g}^{-1} \text{min}^{0.5}$)	0.0797	1.0606

$^{\dagger}\alpha$ = Initial adsorption rate

$^{\ddagger}\beta$ = Initial desorption rate

tK_i = Diffusion Rate constant

Among all the models tested, the PSO model best approximated the TCN adsorption behavior of the SMNZs, as shown in Figures 9 and 10, and distinguished by a high correlation coefficient that is close to unity, as shown in Tables 3 and 4. By contrast, a satisfactory fit could not be obtained for TCN adsorption by UZ. This is likely due to the lack of adsorption indicated by the Q_t vs. time graph, thereby hindering any further analysis. Consistent with the Q_t vs. time graph is the calculated adsorption capacity from the PSO model, which gave the following values in decreasing order: CMZ > AMZ > UZ. Upon comparison of data from kinetics experiments, the TCN adsorption capacities of both SMNZs at 24.33 mg/g (CMZ) and 17.95 mg/g (AMZ) are higher than that of AC (2.02 mg/g) discussed previously.

Kinetic studies provide essential insight into the adsorption mechanism involved, a relevant and necessary parameter in the

design and application of sorption techniques (William Kajjumba et al., 2019). As reviewed in previous literature, the adsorption mechanism that primarily governs unmodified zeolites is ion exchange, owing to their aluminosilicate nature (Elboughdiri, 2020; J. W. Lin et al., 2012; Velarde et al., 2023). The unmodified material tends to give a hydrophilic surface, while monolayer surfactant modification confers an additional hydrophobic surface on the zeolite, thereby introducing another possible rate-limiting step to the adsorption process: hydrophobic partitioning (Bagheri et al., 2024; Sarkar et al., 2021). As TCN is a highly hydrophobic molecule ($\log K_{ow} = 4.79$) (Dhillon et al., 2015), it was likely that favorable adsorbent-adsorbate interactions were optimal for TCN adsorption onto the SMNZs as facilitated by hydrophobic effects (Phuekphong et al., 2021). In contrast, negligible adsorption was observed on the UZs.

4. Conclusion

In this work, SMNZs were successfully synthesized by the modification of PNZs using CPyCl and ARQ, as confirmed by the various characterization techniques. This expands the applicability of surfactant modification onto PNZs beyond clays and other minerals. The adsorption experiments conducted in this study demonstrate that surfactant modification with both surfactants conferred adsorptive capability on the raw zeolite, which had no prior affinity for TCN, with SMNZs exhibiting a significantly higher %TCN removal than UZ. Variation of pH and differences in the surfactants, CPyCl (one-tailed) and ARQ (two-tailed), used in modification did not influence % TCN removal. Adsorption by both adsorbents was best described by the PSO model, which highlights the significance of optimal adsorbent-adsorbate interactions as an integral part of the adsorption mechanism. In comparison to activated carbon, the synthesized SMNZs exhibit great potential as a TCN adsorbent, given their comparable experimental adsorption capacity, lower required adsorbent dose, and fast equilibration determined from kinetic studies. Regeneration and reusability studies may be performed to fully explore their potential as an adsorbent in actual wastewater treatment systems.

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