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Adsorption of Ketoprofen from Octa(aminophenyl) silsesquioxane Nanocomposite

¹Juth Daniel, ²Bala Suleiman, and ³Tanko Mahmoud Mohammed

¹Department of Science Laboratory Technology, Federal Polytechnic Mubi, Adamawa State,

²Department of Chemistry, Federal University of Agriculture Mubi, Adamawa State

³Department of Biomedical and Pharmaceutical Technology, Federal Polytechnic Mubi, Adamawa State

Abstract

Silsesquioxanes are organosilicon compounds with the general chemical formula $(\text{RSiO}_{1.5})_n$, where R represents the vertex substituent attached to the polyhedral framework and may comprise hydrogen, alkyl, alkene, aryl, or arylene groups. Among the different silsesquioxane architectures, Polyhedral Oligomeric Silsesquioxane (POSS) has attracted considerable research interest because of its well-defined inorganic-organic structure. POSS molecules commonly possess an inorganic T_8 cubic core consisting of silicon-oxygen linkages with the molecular composition $\text{R}_8\text{Si}_8\text{O}_{12}$. In this study, Octa(aminophenyl) silsesquioxane (OAPS) was carefully synthesized through a sequential chemical modification of POSS. The synthesis involved the nitration of POSS to produce Octa(nitrophenyl) silsesquioxane (ONPS), followed by reduction of the nitro-functionalized intermediate to obtain the target product, Octa(aminophenyl) silsesquioxane (OAPS). The synthesized material was characterized using Powder X-ray Diffraction (PXRD), Fourier Transform Infrared (FTIR) spectroscopy, and Nuclear Magnetic Resonance (NMR) spectroscopy. NMR characterization included ^1H , ^{13}C , and ^{29}Si analyses to investigate the chemical environment and structural characteristics of the synthesized material. Following synthesis and characterization, the resulting OAPS material was subjected to adsorption studies to evaluate its potential application as an adsorbent. The adsorption investigation demonstrated promising performance, indicating that the synthesized material is capable of removing pharmaceutical and anti-inflammatory drugs. These findings demonstrate the potential applicability of functionalized Polyhedral Oligomeric Silsesquioxane materials for adsorption-based removal of pharmaceutical contaminants and provide a basis for their further investigation in environmental remediation and treatment applications.

Keywords

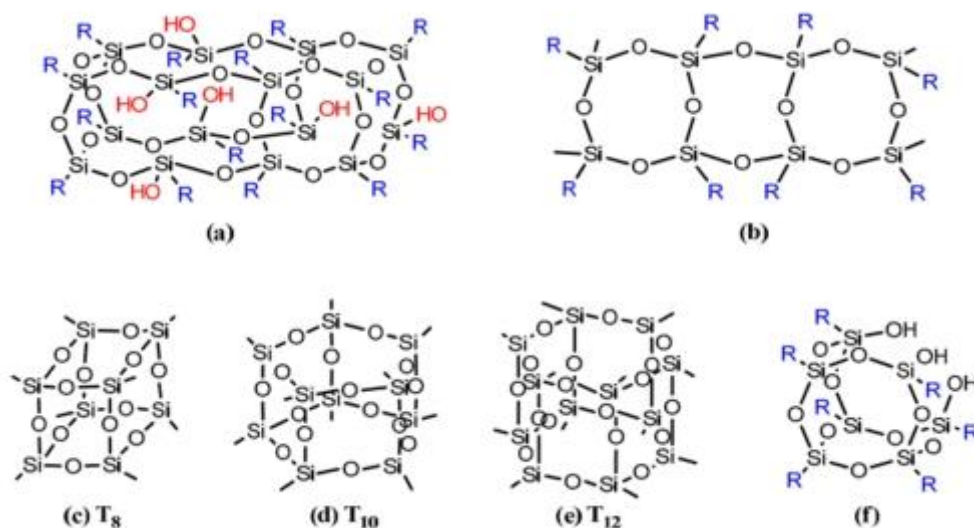
Polyhedral Silsesquioxane,
Reduction,
Adsorption,
Inflammatory



Introduction

Study on the chemistry of silesquioxanes has been carried out for more than 50 years. However, in recent times, there has been an explosion of interest in this area of (Zhou, Ye, & Xu, 2017; Zhu & Zhang, 2017). Silesquioxanes refer to the compounds that have a chemical formula of $(\text{RSiO}_{1.5})_n$. R denotes the vertex group in polyhedral. This can be hydrogen, alkyl, alkene, aryl, arylene (Bala, Abdullah, Tahir, & Abdul Rahman, 2022; Suleiman & Mohammed, 2024). The most abundant structure being investigated is that of Polyhedral Oligomeric Silesquioxane (POSS) molecules comprising an inorganic core of T8 cubic structure made up of silicon-oxygen linkages ($\text{R}_8\text{Si}_8\text{O}_{12}$). In this article, "T" and "Q" are used in their

usual notations of the silicon NMR literature where T and Q are used for silicon atoms having three and four oxygen atoms attached to them, respectively. Unlike traditional organic molecules, the POSS materials are volatile, odourless, and environmentally safe. When POSS functionalities are incorporated into the polymeric matrix, the mechanical properties of the polymer and the overall polymer properties improve greatly (Bala et al., 2022; Kausar, 2017). These molecules, which exhibit a high degree of symmetry, tend to have an extremely nano size ranging between 1 to 3 nm in diameter when the vertex group is included, and these can be considered some of the smallest silica particles (Mohammed, 2026; Suleiman & Mohammed, 2024).



Scheme 1: Chemical structures of silesquioxane. (I) Non-caged silesquioxane structures; (a) random structure, (b) ladder; (II) Caged silesquioxanes: (c) T₈, (d) T₁₀, (e) T₁₂ structures. (f) partial cage.

The researcher's attention has been increasingly shifting to a unique category of contaminants referred to as "emerging pollutants" which were not part of the regulations (non-priority pollutants) such as the pharmaceutical and personal care products (PPCPs), which have emerged as a novel conservation threat of concentration (Jennifer, Abdallah, & Harrad, 2017). The PPCPs have attracted global attention owing

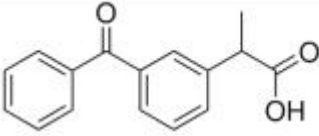
to their massive consumption (Chu et al., 2017; Gadipelly et al., 2014). "Emerging pollutants" can be defined as those pollutants that enter or form in large amounts in the environment with some degree of resistance and exhibit some negative effect on the species (Bala et al., 2022; Hu, Yan, Ge, & Gao, 2018; Pi et al., 2018). One of the interesting characteristics of some of the pollutants that can lead to

such pollution is that to have any adverse effect on the environment, they do not necessarily have to be permanent in the atmosphere. This is due to the fact that their significantly faster rate of degradation and conversion can compensate for the continuous introduction of these pollutants in the environment; sometimes through waste managements (Ma et al., 2018; Pan et al., 2013; Song, Sun, Aguila, & Ma, 2019).

Pharmaceuticals that are frequently utilized include anti-inflammatory drugs (AIDs) (I. Ahmed & Jhung, 2017; Mohammed, 2026). Because of their analgesic, anti-inflammatory, and antipyretic qualities, these medications are used to treat conditions in both humans and animals (Feng et al., 2013). AIDs can stay in the aqueous phase for extended periods of time due to their great hydrophilicities and water

stabilities (Abdelnaby et al., 2018; Gendy et al., 2021). Moreover, both AIDs and the most often discovered medications in aquatic environments are highly resistant to biological degradation (Du et al., 2016; Hou et al., 2015). Among other growing water contaminants, ketoprofen [2-(3-benzoylphenyl) propanoic acid, or KTP] is frequently used as an analgesic and antipyretic (Baccar, Sarrà, Bouzid, Feki, & Blázquez, 2013; Suleiman & Mohammed, 2024). Urine and improper disposal are two ways that KTP can end up in water. Moreover, both AIDs and the most often discovered medications in aquatic environments are highly resistant to biological degradation (Feng et al., 2013; Im et al., 2013). The physical and molecular characteristics of KTP are displayed in Table 1.

Table 1: Chemical structure and properties of KTP

	
Molar mass (g·mol ⁻¹)	254.3
pKa	4.5
Solubility in water (mg·L ⁻¹)	51.0

(Mohammed, 2026; Song & Jhung, 2017; Suleiman & Mohammed, 2024)

Methodology

Octa(phenyl)silesquioxane (OPS), fuming nitric acid, Tetrahydrofuran (THF), Hydrazine hydrate, Hexane, Ethylacetate, Dimethylacetamide (DMAc), Mesitylene, 1,4-dioxane, Dimethylformamide (DMF), Methanol, Ethanol, Acetonitrile, Chloroform, Chlorobenzene, an Acetic acid, Iron (III) chloride hexahydrate (catalyst), Magnesium

sulphate (anhydrous), Charcoal powder, Pd/C (catalytic hydrogenation), Celite etc.

Synthesis of Octa (nitrophenyl) silesquioxane (ONPS)

ONPS, (C₄₈H₃₂Si₈N₈O₂₈) will be produced with a slender reformation (Afiq, Musa, Yin, & Savory, 2010; Bala et al., 2022; Suleiman &

Mohammed, 2024). OPS, ($C_{48}H_{40}Si_8O_{12}$) 5g (4.84 mmol), will be supplemented to a 30 mL fuming nitric acid. Then, mix at 0°C for additional 30 min, then agitated at room temperature for further 20 h. The combination will further be dispensed onto 250 g of an ice bag, upon filtration. A quite faintly yellow precipitate will be treated with water (around 100 mL five (5) times until pH approximately 6), then ethanol (100 mL 3 times). The residue will be collected and dried in an oven at ~ 80 °C to eliminate the remaining moisture.

Synthesis of

Octa(aminophenyl)silesquioxane (OAPS)

In a 250 mL 3-necked round-bottom bottle fixed with a mechanical stirrer and a condenser, 3g of ONPS (2.60 mmol), 120 mg $FeCl_3 \cdot 6H_2O$ (catalyst), 1.5 g charcoal powder will be supplemented. The flask will then be accompanied with distilled THF (40 mL), and the solution will be stirred and heated to 60 °C, under N_2 . Hydrazine hydrate (13 mL) will be add-on dropwise into the admixture. The reaction will be advanced for 5 h, and then the mixture will be ventilated and purified through celite, the filtrate will be merged with 25 mL ethyl acetate, then washed three times with distilled water. The organic layer will be desiccated over Na_2SO_4 and will be dispensed into 250 mL hexane, the precipitate formed will be accrued by filtration. The substance will be redissolved in range of 15 mL THF and 25 mL ethyl acetate and reprecipitated into 250 mL hexane. The off-white precipitate OAPS ($C_{48}H_{48}Si_8N_8O_{12}$) will be vacuum-dried for 48 h. (Bala et al., 2022; Mohammed, 2026; Zhang, Xu, Yu, & Si, 2006).

Adsorption Experiments

Serial dilutions of ketoprofen (KTP) (50 mgL⁻¹) were made by dissolving a 90:10 v/v water-methanol solvent. The pH level was adjusted to 7.0 by suggesting the necessary volume of

0.1M NaOH aq. solution (taking into account the normal pH of river and rainwater) (Bala et al., 2022; Bhadra & Jung, 2017; Sarker, Song, & Jung, 2018a). The reference solution was serially diluted to create KTP solution at the basic concentrations (5-100 mgL⁻¹).

The concentration of KTP was ascertained by establishing the solution's absorption spectra at 262 nm using a UV spectrophotometer (UV-1650, Shimadzu). The adsorbent was dried at 100 °C for 12 hours in a vacuum oven to distribute water prior to the adsorption. After adding the adsorbent (5.0 mg) to the adsorbate mixture (25 mL, pH 7.0), the mixture was agitated in a magnetic stirrer at a steady speed of 250 rpm for one to five hours at 25 °C. The mixture was sieved on a polytetrafluoroethylene syringe filter (hydrophobic, 1 ml) after the adsorption analysis.

The powder fraction of adsorbates in the solution was analysed using the UV spectrophotometry spectrum. Afterward, by comparing a volume of 0.1 M HCl (or NaOH) solution to the solution, the impact of solution pH on KTP adsorption on Adsorbate was investigated. Twenty milligrams of the administered COFs were added to ethanol to conduct the regeneration investigation.

Results

Characterization of adsorbent

The as-synthesized nanomaterial was studied utilizing multiple spectroscopic procedures such as FTIR, PXRD, NMR (1H ^{13}C and ^{29}Si). FTIR Spectroscopy exhibited that the ONPS and OAPS were successfully synthesized as shown in **Figure 1a**. $\nu N=O$ symmetry and asymmetry bonds appeared in ONPS at 1344 and 1537 cm⁻¹ respectively. Both peaks were reduced and new broad peaks occurred at 3352 and 3224 cm⁻¹ which

were assigned to ν N-H bonds. Si-O-Si bond were observed at 1092 and 1064 cm^{-1} for ONPS and OAPS respectively. This is in conformity with the literature reported (Bala et al., 2022; Zhang et al., 2006). PXRD pattern of the OAPS composite agreeing with the calculated display, demonstrating that the nanomaterial was effectively produced. OAPS exhibited a strong diffraction peak at 7.8° , this

diffraction peak could be attributed to the diameter of the OAPS cage (**Figure 1b**). The materials demonstrated obvious strong peaks at below 10 degrees (< 10). Furthermore, a broad peak was observed at 21.7° in the OAPS diffraction pattern, which was associated with the presence of amine substitutional (Bala et al., 2022; Mohammed, 2026; Zhang et al., 2006).

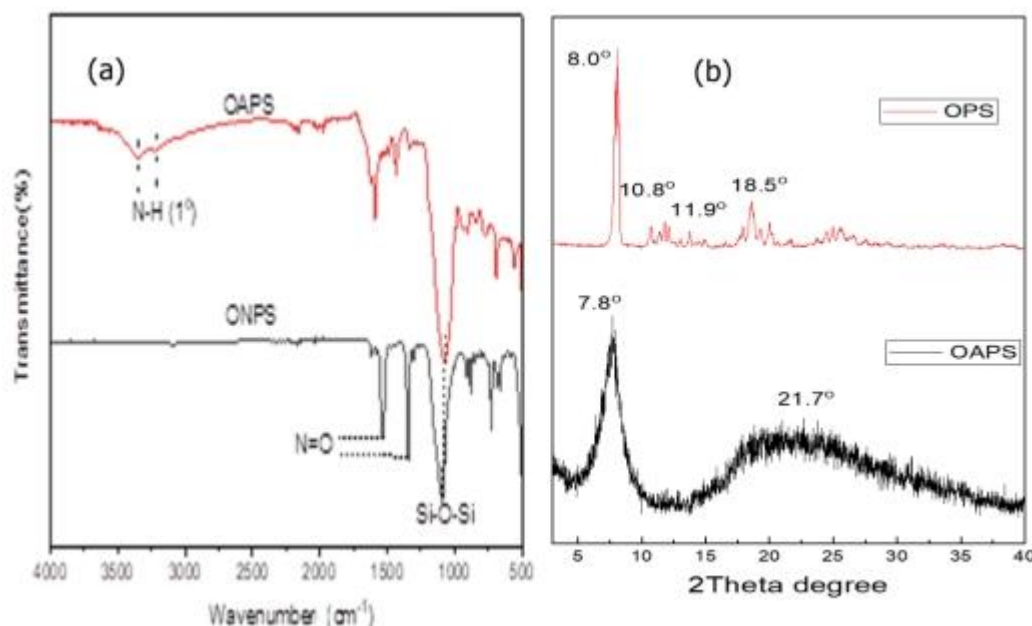


Figure 1. (a) FT-IR Spectroscopy and (b) PXRD spectra of the material synthesized

In the ^1H NMR spectrum of OAPS, no peak at 8–9 ppm corresponding to the protons in aromatic groups of ONPS was observed, while the aromatic hydrogens of OAPS were detected at higher magnetic field (7.8–6.0 ppm) due to the electron donor properties of amino groups on the phenyl rings as exhibited in **Figure 2a**. This is in agreement with the study documented (Bala et al., 2022; Mohammed, 2026). Solid State ^{13}C -MAS NMR of OAPS were analysed which shown that the polyhedron Oligosilsesquioxane

building blocks were retained during the synthesis. The ^{13}C -NMR (**Figure 2b**) also proved the process, as signals from the trends assigned to ONPS were completely replaced by the new peaks of OAPS cluster at higher region 118.0–152.9 ppm at the end of the synthesis. Precisely, four respective peaks belong to the aromatic carbon environments emerged at 118.05, 132.39, 147.27 and 152.93 ppm were detected. This illustrates that the cage framework of polyhedral Oligosilsesquioxane is conserved

throughout processing. Same situations were affirmed by (Commun et al., 2001; Liu et al., 2019; Miao et al., 2019). Additionally, ^{29}Si solid state NMR spectrum for OAPS was done to further validates the consistency of the oligomeric silesquioxane cage framework in the formulation as demonstrated in **Figure 2c**. The result obtained obviously showed

that the cage structure of the polyhedral silesquioxane is sustained during the production as shown in respect of OAPS peak positions at -79 and -69.6 ppm. This is in agreement with the documented research studies by conventional method using Pd/C catalyst (Bala et al., 2022; Mohammed, 2026; Ni & Zheng, 2004; Ren et al., 2010).

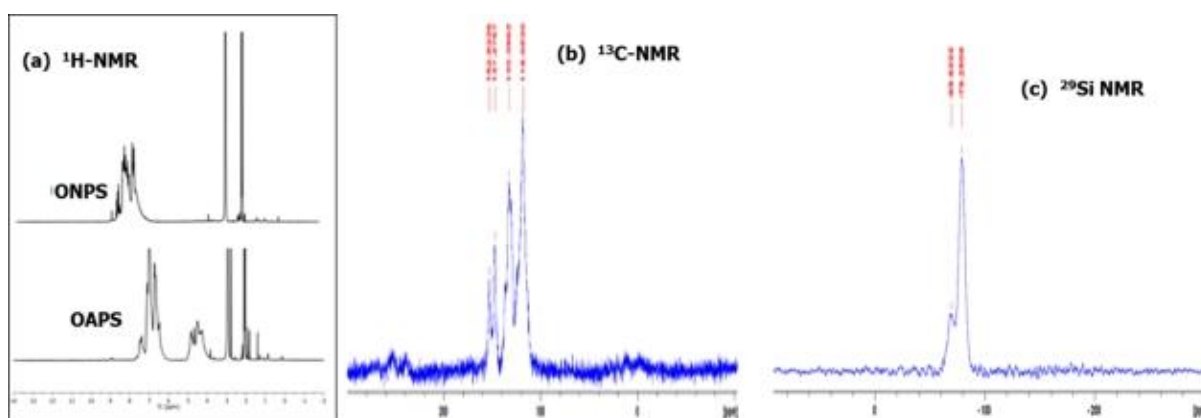


Figure 2. (a) ^1H -NMR spectra of ONPS and OAPS (b) Solid state ^{13}C -NMR spectra of OAPS and (c) ^{29}Si -NMR of the OAPS nanomaterial

Figure 3a, b, c, and d illustrate the ketoprofen effect of pH, adsorption capacity, removal efficiency, and regeneration study. Since KTP's acid dissociation constant (pK_a) is 4.5, adsorption of KTP is regarded as a minimal acidic (Bala et al., 2022; Hao et al., 2017; Sarker, Song, & Jhung, 2018b; Sarker et al., 2018a; Xue et al., 2017). Therefore, in $\text{pH} < 4.5$ and $\text{pH} >$

4.5, respectively, it exists in the neutral and deprotonated/anionic forms. The pH range of the substance under study was 4-14. Additionally, the materials provided a gradual decrease with an increase in pH rate equilibrium time (q_t) of 260 minutes, the obtained data showed that there is firm exclusion with an increase in pH to 9.

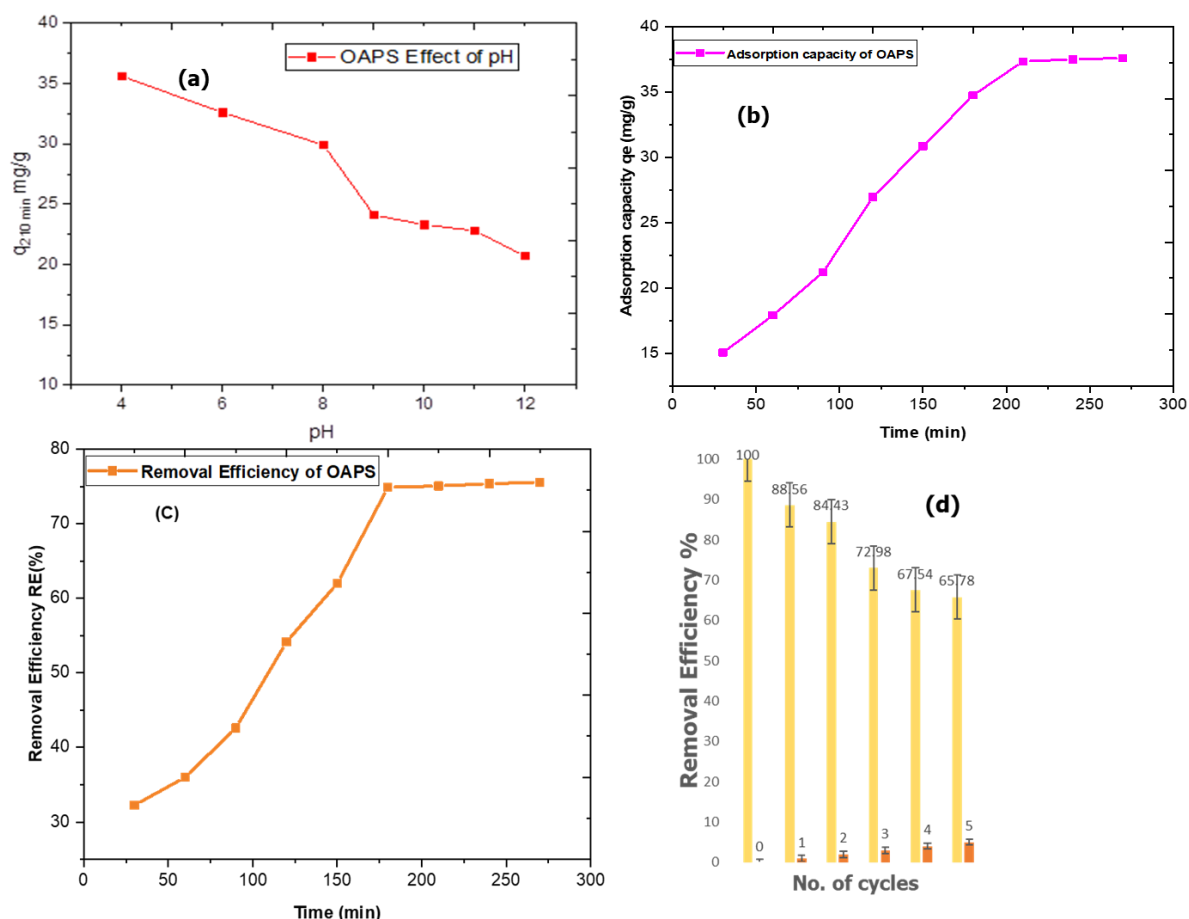


Figure 3. (a) Effect of pH, (b) Adsorption capacity (c) Removal efficiency and (d) regeneration study on the absorbed amount of ketoprofen over synthesized OAPS.

The nanocomposite exhibited certain escalations as early as 32 minutes. The material established a steady and slower increases of removal after 2.5 hours, Ketoprofen removal was realized of nearly 50 % after 2 hours as in Figure 2c. Similarly, the adsorption capacity (q_e) displayed a maximum adsorption capability experimental (q_{max}) at 38 and computed (q_{max}) 15.36 for OAPS. Similarly, the adsorption capacity (q_e) displayed a maximum adsorption capability experimental (q_{max}) at 22.5 and computed (q_{max}) 15.365 nanomaterial respectively. The nanoparticle suffered a nearly uniform drifts of removal proficiencies down to

65.86 and 62.43 % after 5 cycles respectively (M. J. Ahmed, 2017; Bhadra, Seo, & Jhung, 2016; Suleiman & Mohammed, 2024). After five cycles, the nanoparticle's removal ability floated almost uniformly to 65.86 and 62.43%, respectively. Adsorption power was conceded (Mohammed, 2026; Sarker et al., 2018a, 2018b).

Conclusion

The nanocomposite was successfully synthesized via the nitrating of OPS to form ONPS and then reducing ONPS to yield OAPS. Analytical techniques such as FTIR, PXRD, and NMR were used to further

examine the substance and attest by documents available in the literatures. Adsorption capacity, removal efficiency, adsorption isotherm, and regeneration of the adsorbed material were measured. After 260 minutes, the nanomaterials showed a significant degree of no strict decrease in the adsorption efficacy with an increase in the number of recycles, indicating that the material can be regenerated multiple times without remarkable adsorbates. Therefore, based on its philosophical deep assembly and adsorption reusability investigation, COF synthesized may be suggested in the future for the adsorption efficacy of pharmaceutical waste.

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