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Comparative Analysis of Commercial and Synthesized Molybdenum Disulfide for Progesterone Removal in Water Treatment

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Abstract: *Water contamination by Endocrine Disrupting Chemicals (EDCs), such as progesterone, is a significant environmental concern. This study uniquely investigates the efficacy of synthesized molybdenum disulfide (S-MoS₂) compared to commercial molybdenum disulfide (C-MoS₂) for progesterone removal from water. Batch experiments evaluated the removal efficiency of both types under various dosages and pH conditions. Preliminary results indicate that S-MoS₂ achieves 12.8% higher removal efficiency than C-MoS₂. This results of the enhanced properties of S-MoS₂ attributed to its synthesis. Dosage experiments showed a 10.7% improvement at an optimal dosage of 20 mg/L (87.8% vs. 79.3%). Additionally, pH variation experiments revealed the highest removal efficiency at pH 7, with a 14.8% improvement (89.3% vs. 77.8%). These findings highlight the superior performance of S-MoS₂, demonstrating its potential for effective progesterone removal in water treatment applications. This research is the first of its kind to compare synthesized and commercial MoS₂ for this purpose, marking a significant advancement in the field of water treatment.*

Keywords: Progesterone; Endocrine-disrupting chemical; Molybdenum disulfide; Nanomaterials; Water treatment;

1. INTRODUCTION

Water contamination by Endocrine Disrupting Chemicals (EDCs) has emerged as a significant environmental concern, according to the World Health Organization [1]. Among these EDCs, progesterone (PG), a naturally occurring steroid hormone, has been detected in various water bodies around the world. Its presence poses a potential risk to human health by affecting the reproductive health, cardiovascular system, cognition, skeletal, and immune system [2]. Additionally, PG contamination can disrupt aquatic ecosystems, impacting the health and biodiversity of aquatic life [3].

Despite these negative impacts, PG-based drugs are among the most prescribed medications for women [4]. PG's main functions in the body are to regulate menstruation and support pregnancy. It is also prescribed as an oral contraceptive and for hormone replacement therapy [5]. Over 100 million women currently use oral contraceptives containing PG [6].

The World Health Organization (WHO) projects this number will increase to approximately 800 million by 2030 [7]. Correspondingly, the global market for PG is expected to grow to an estimated value of \$2.1 billion by that year [8]. This significant increase in usage could lead to heightened levels of PG contamination in water sources, necessitating improved water treatment technologies to mitigate the potential environmental and health impacts [9].

Molybdenum-based materials have shown promising applications in a wide range of fields such as catalysis and photocatalysis, transistors and sensors, energy storage, dry lubrication, and even environmental remediation [10][11][12]. Due to its unique properties, molybdenum disulfide (MoS₂) has been extensively studied in recent years and shows great promise in the field of environmental remediation and water treatment [13]. However, the performance of commercially available MoS₂ (C-MoS₂) compared to synthesized MoS₂ (S-MoS₂) customized for the removal of PG in water has not been thoroughly

examined. This study aims to fill this gap by conducting a comparative analysis through batch experiments of C-MoS₂ and S-MoS₂ for the removal of PG from water under various pH conditions and MoS₂ dosages.

2. MATERIALS AND METHODS

2.1 Materials

Synthetic PG (C₁₂H₃₀O₂, 98%) was procured from Tokyo Chemical Industry (TCI, Japan) to prepare the stock solution. Ammonium molybdate tetrahydrate ((NH₄)₆Mo₇O₂₄ · 4H₂O, 99%) was purchased from FUJIFILM Wako Pure Chemical Corporation, Japan, for the synthesis of MoS₂ nanoparticles. Thiourea (CH₄N₂S, 99%) was obtained from Sigma-Aldrich, USA, for the synthesis of MoS₂ nanoparticles. The C-MoS₂ (MoS₂, 99%) that was used for the comparative analysis was purchased from Kojundo Chemical Laboratory Co. Ltd, Japan. Ethanol (C₂H₅OH, 99.5%) was purchased from FUJIFILM Wako Pure Chemical Corporation, Japan, to dissolve the PG for stock solution preparation. Sodium hydroxide (NaOH, 97%) was acquired from JENSEI, Japan, for pH adjustment. Hydrochloric acid (HCl, 35-37%) was purchased from FUJIFILM Wako Pure Chemical Corporation, Japan, for pH adjustment.

2.2 Preparation of stock solution

20 mg of PG was dissolved in 10 mL of ethanol. Then, the PG-ethanol solution was transferred into a 1 L volumetric flask containing ultrapure deionized water (UPDI). The solution was mixed at 1000 rpm and 25°C to obtain 20 mg L⁻¹ solution of PG.

2.3 Synthesis of molybdenum disulfide

MoS₂ was synthesized following the hydrothermal method described by Zhou et al. [14]. In this method, 0.6g of Ammonium molybdate tetrahydrate and 1.2g of thiourea were dissolved in 70 mL of UPDI while stirring vigorously. The mixture was then transferred to a 100 mL Teflon-lined stainless-steel autoclave and kept at 200°C for 20 hours. The autoclave was subsequently removed and set aside to cool to ambient temperature. Upon cooling, the mixture was placed into a centrifuge, and the precipitate was collected and washed three times with anhydrous ethanol and UPDI. The precipitate was placed in a vacuum dryer at 70°C overnight to dry. The crystalline structure and chemical composition of S-MoS₂ and C-MoS₂ were evaluated using X-ray diffraction analysis (XRD; Smartlab, Rigaku, Japan) [15].

2.4 Batch experiments

To investigate the effects of dosage, specific masses of 1 mg, 5 mg, and 10 mg of both C-MoS₂ and S-MoS₂ were carefully measured on a weighing dish using an analytical scale. The measured masses were then transferred into a vial, followed by the addition of 5 mL of UPDI water, which was sprayed onto the weighing dish to clean off any residue and then decanted into the vial. The vials were then ultrasonicated for 30 minutes to promote the distribution of nanomaterial throughout the water. While the ultrasonication took place, the batch experiment was set up using 50 mL Erlenmeyer flasks. 45 mL of stock solution was decanted into the flasks. After 30 minutes, the vials were removed from the ultrasonic bath, and their contents were poured into the Erlenmeyer flasks containing the stock solution. The vials were then backwashed with stock solution as needed to ensure complete transfer of material. The flasks were subsequently positioned on magnetic stirrers with a rubber stopper and agitated at 1000 rpm and 25°C. After a designated contact period between MoS₂ and the contaminant, the stirrer was disabled for 5 minutes to allow some of the particulates to settle prior to sample extraction. Samples were extracted using a syringe, and dispensed through a 45 µm filter (ADVANTEC Dimic, Japan), and collected in sample vials for analysis. The sampling procedure was repeated as needed to obtain multiple samples for comprehensive analysis [16].

To investigate the effects of pH on the removal efficiency, sodium hydroxide (NaOH) and hydrochloric acid (HCl) were used to adjust the pH of 45 mL of PG stock solution. The pH of the solutions was carefully modified to 3, 7, and 10 using a Horiba LAQUA D-210P. Next, 5 mg of both C-MoS₂ and S-MoS₂ were measured, ultrasonicated and decanted into the 50 mL flasks, similar to the aforementioned dosage procedure. The pH was also recorded at the end of the experiment to ensure precision.

2.5 Analytical instruments

After the PG solution was sufficiently treated with MoS₂ nanoparticles, it was ready for analysis. The residual concentration of PG was measured using a UV-vis spectrophotometer (Shimadzu UV-1280, Japan) at a peak wavelength of approximately 250 nm [17]. The UV-vis spectrophotometer was calibrated using several standard PG solutions with concentrations ranging from 0.25 to 20 mg L⁻¹. The following calibration curve was derived [18]:

$$Y = 38.205X - 0.3747 \quad (1)$$

Where X and Y represent the concentration of PG (mg L⁻¹) and the absorbance value measured by the UV-vis spectrophotometer at 250 nm, respectively.

2.6 Performance evaluation

The effectiveness of the MoS₂ nanoparticles was evaluated by determining the final removal efficiency (RE) using the following equation [19]:

$$RE(\%) = \frac{(C_i - C_f)}{C_i} \times 100\% \quad (2)$$

Where C_i and C_f represent the initial and final concentrations in mg L⁻¹, respectively.

3. RESULTS AND DISCUSSION

3.1 XRD and structural characterization

The synthesized MoS₂ was characterized by X-ray diffraction (XRD), and the broad diffraction peaks are represented in Fig. 1.

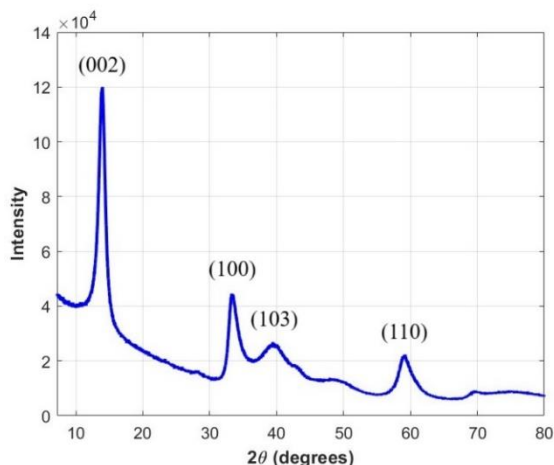


Fig. 1. XRD pattern for S-MoS₂

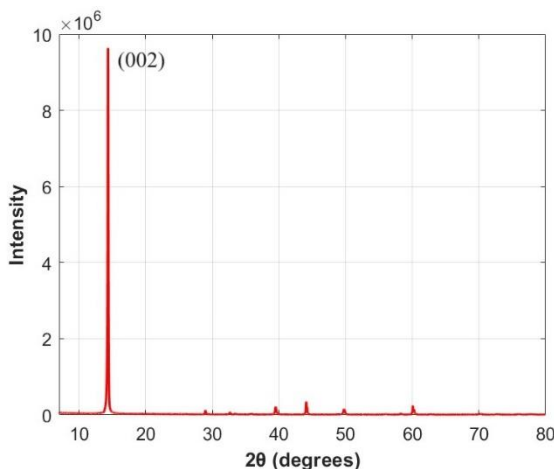


Fig. 2. XRD pattern for C-MoS₂

The XRD indicates nanoscale MoS₂ in the (002) lattice plane, as evidenced by the 14.4° peak. This illustrates the single-layer stacking of crystalline MoS₂ along the c-axis, connected by weak Van der Waals bonding and is associated with large surface area [17][18][19]. This can also be seen in Fig. 2. Additionally, the intensity of the (002) peak for C-MoS₂ is notably higher compared to S-MoS₂, suggesting a greater degree of crystallinity or a higher concentration of the crystalline phase in C-MoS₂. The minor peaks observed at higher angles correspond to other crystal planes, but their intensities are much lower compared to the (002) peak. In Fig. 1, we can observe another peak at approximately 32.6° in the XRD pattern, which coincides with the (100) plane in the hexagonal structure of the MoS₂ [21]. Moreover, another peak can be found at 39.5°, and this is associated with the (103) plane, which has been reported to further confirm the hexagonal phase structure of MoS₂ [23]. The last distinct peak can be seen at 49.8°, which corresponds with the (110) plane. Overall, these peaks confirm the hexagonal structure, high crystallinity, nanoscale size, and purity of the synthesized molybdenum disulfide [23].

3.2 Preliminary experiment

The preliminary experiment aimed to evaluate the removal efficiency of C-MoS₂ compared to S-MoS₂ in removing PG from water. As shown in Fig. 3, the C-MoS₂ achieved a removal efficiency of 53.9%. In contrast, the S-MoS₂ exhibited a higher removal efficiency of 60.8%.

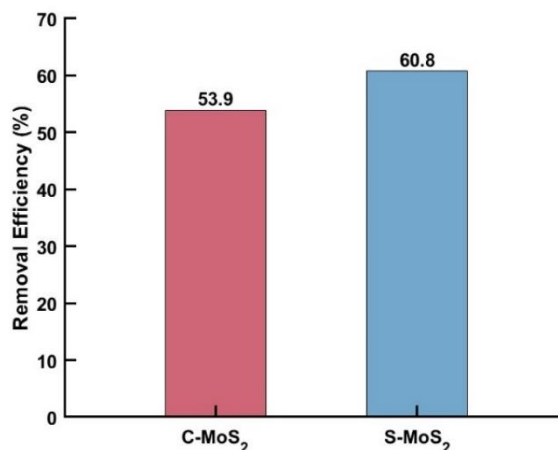


Fig. 3. Preliminary experiment using C-MoS₂ and S-MoS₂ for the removal of PG in water. Conditions: Contact time = 1 hour, MoS₂ dosage = 100 mg L⁻¹, temperature = 25°C, pH = 7, initial PG concentration = 20 mg L⁻¹, mixing rate = 1000 rpm.

The S-MoS₂ demonstrated superior performance in PG removal compared to C-MoS₂, sparking significant interest and catalyzing further investigations into optimizing its performance. The enhanced performance of S-MoS₂ could be attributed to factors such as increased surface area, enhanced adsorption capacity due to surface defects, or beneficial discrepancies in the material's crystallinity [14]. To explore these characteristics and gain insight into the removal mechanisms involved, further investigations were conducted.

3.1 Effect of nanomaterials dosage

The dosage of nano-adsorbents typically plays a significant role in the removal of contaminants [24][25]. As demonstrated in Table 1, three dosages of MoS₂ were utilized to evaluate their effects on removing PG from water.

Table 1. Dosages of C-MoS₂ and S-MoS₂ investigated.

MoS ₂ mass (mg)	Dosage (mg/200ml)	Dosage (mg/ L)
4	4	20
20	20	100
40	40	200

The results of varying dosages on the removal of PG solution with a concentration of 20 mg/L after 1 hour are presented in Fig. 4. It is evident that S-MoS₂ consistently outperformed C-MoS₂ at all dosages tested. Notably, at the optimal dosage of 20 mg/L, the removal efficiency using S-MoS₂ was 87.8%, compared to 79.3% with C-MoS₂, representing a 10.7% improvement. This comparative analysis highlights the superior performance of S-MoS₂ over C-MoS₂ in the removal of PG from water.

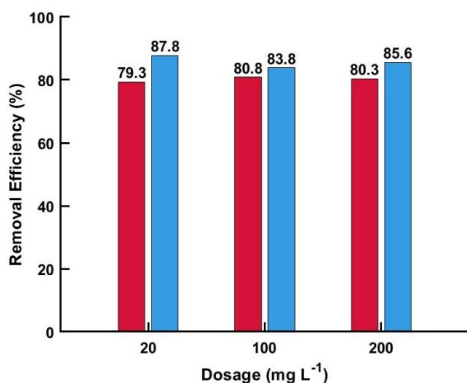


Fig. 4. Effects of dosage on the removal efficiency of PG using C-MoS₂ (red) and S-MoS₂ (blue). Conditions: Contact time = 1 hour, MoS₂ dosage = 20-200 mg L⁻¹, temperature = 25°C, pH = 7, initial PG concentration = 20 mg L⁻¹, mixing rate = 1000 rpm.

3.2 Effects of pH

The pH of a solution significantly impacts the removal efficiency of contaminants by affecting the ionization state of the contaminants and the surface charge of adsorbent, thereby influencing adsorption capacity [18][26]. The influence of varying pH conditions on the performance of C-MoS₂ and S-MoS₂ in removing PG was evaluated after 1 hour of contact time, and the results are represented in Fig. 5.

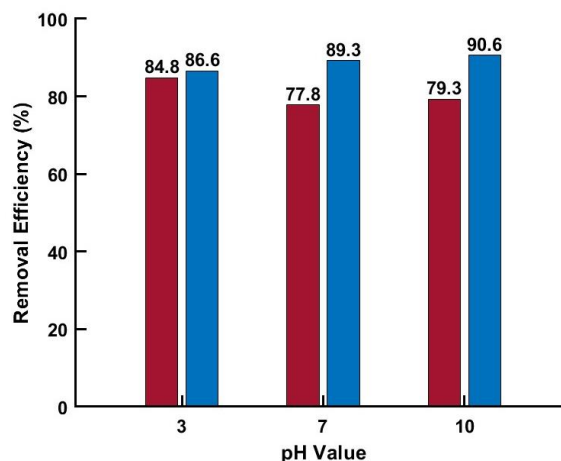


Fig. 5. pH-dependent removal efficiency of PG using C-MoS₂ (red) and S-MoS₂ (blue). Conditions: Contact time = 1 hour, MoS₂ dosage = 20 mg L⁻¹, temperature = 25°C, pH = 3-10, initial PG concentration = 20 mg L⁻¹, mixing rate = 1000 rpm.

As demonstrated, S-MoS₂ has considerably outperformed C-MoS₂ across all tested pH levels. Notably, at a neutral pH of 7, S-MoS₂ achieved a removal efficiency of 89.3%, significantly higher than the 77.8% achieved by C-MoS₂, indicating a substantial improvement of 14.8 percentage points. This superior performance of S-MoS₂ is consistent across acidic (pH 3) and alkaline (pH 10) conditions, highlighting its robust efficiency in diverse pH environments. The variation in performance can be attributed to the enhanced surface area and adsorption capacity of S-MoS₂, as well as its increased surface defects and optimized crystallinity, which improve the interaction mechanisms between the adsorbent and the contaminants [27]. These results underscore the potential of S-MoS₂ in addressing the diverse pH conditions encountered in real-world water treatment applications [24].

3.3 Enhanced adsorption mechanism of S-MoS₂

The superior performance of S-MoS₂ can be attributed to its distinct synthesis method, which results in

surface defects that increase the surface area and enhance interactions with the PG molecules [14]. These defects, vacancies, and grain boundaries on MoS₂ act as activation sites, increasing adsorption energy and thereby improving PG adsorption [28]. Additionally, the hydrophobic nature of PG allows for better interaction with the hydrophobic regions of MoS₂, promoting mutual affinity [29]. PG's functional groups, such as carbonyl, can form hydrogen bonds with sites on the MoS₂ surface, further enhancing PG uptake [30][31]. Incorporating oxygen into MoS₂ has been shown to improve adsorption properties by enhancing complexation with sulfur atoms on the MoS₂ surface, increasing binding sites, and building stronger interactions [32]. Moreover, MoS₂ may facilitate redox reactions, degrading compounds like PG into less harmful forms [33]. It is likely that a combination of these mechanisms contributes to the efficient removal of PG from water.

4. CONCLUSION

This study evaluated the efficiency of commercially available MoS₂ and synthesized MoS₂ in removing PG from water under various conditions. The synthesized MoS₂ outperformed the commercial counterpart, achieving a 12.8% higher removal efficiency in preliminary experiments. The dosage-dependent experiments showed superior performance for S-MoS₂, with an optimum dosage of 20 mg/L achieving an 87.8% removal efficiency. Furthermore, pH variation experiments demonstrated that S-MoS₂ maintained higher removal efficiency at all tested pH levels, particularly excelling in neutral and alkaline conditions, with improvements of 14.8% at pH 7 and 14.2% at pH 10. Overall, the S-MoS₂ exhibits superior performance in PG removal from water compared to C-MoS₂ across various dosages and pH levels. These results highlight its potential for optimizing PG removal in water treatment applications, making it a promising material for further research and development.

5. ACKNOWLEDGEMENT

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