# **Review Paper**

# A Review of the Sonication-Assisted Exfoliation Methods for MoX<sub>2</sub> (X: S, Se, Te) Using Water and Ethanol

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Two-dimensional transition metal dichalcogenides (MoX<sub>2</sub>, where X = S, Se, Te), have been the research hotspot over the past decade. The sonication-assisted liquid-phase exfoliation method is suitable for the mass production of MoX<sub>2</sub> in practical applications. Water and ethanol, rather than organic solvents, are increasingly chosen for liquid-phase exfoliation method due to their non-toxic, environmentally friendly properties. However, a systematic review of the method for MoX<sub>2</sub> preparation using water and ethanol is lacking. In this paper, recently published work on the sonication-assisted exfoliation method for MoX<sub>2</sub> preparation using water and ethanol is summarized. Three key parameters are focused on: solvents selection, sonication power, and sonication time. Finally, the application of MoX<sub>2</sub> flakes and the future outlook of the sonication-assisted liquid-phase exfoliation method using water and ethanol are presented. The review aims to provide guidance on exfoliating MoX<sub>2</sub> using the sonication-assisted exfoliation method with water and ethanol.

**Keywords:** two-dimensional transition metal dichalcogenides; sonication-assisted liquid-phase exfoliation; water; ethanol.



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# 1. Introduction

Since graphene was discovered by NOVOSELOV et al. (2004), two-dimensional (2D) materials have become a hotspot in materials research. So far, various 2D materials have been studied, including the insulator boron nitride (BN), semiconductor transitional metal dichalcogenides (TMDs), magnetic materials such as  $CrX_3$  (I, Br),  $Fe_3GeTe_2$ , and the topological insulator Bi<sub>2</sub>Se<sub>3</sub>, among others. TMDs MX<sub>2</sub> (where M = Mo, W, and X = S, Se) shows potential in many applications due to its excellent physical properties. For example, MoS<sub>2</sub>-based field-effect transistors (FETs) exhibit a  $\sim 10^8$  on/off ratio with mobility ~200  $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$  (RADISAVLJEVIC *et al.*, 2011). Monolayer  $MoS_2$  also demonstrates a strong photoluminescence effect due to the indirect-to-direct bandgap transition (from 1.9 eV to 2.2 eV) from bulk to monolayer (MAK *et al.*, 2010). Actually, monolayer  $MoS_2$  exists in three distinct phases: the semiconductive 2H phase, the metallic 1T phase, and the 1T' phase. The 2H-MoS<sub>2</sub> phase holds potential development for applications in valleytronics. On one hand, 2H-MoS<sub>2</sub> displays a novel valley degree of freedom due to broken inversion symmetry. On the other hand, its valley and spin degrees of freedom are coupled due to spin-orbit splitting (XIAO *et al.*, 2012). MoSe<sub>2</sub> is more conductive with respect to MoS<sub>2</sub> due to selenium (Se) atoms being more conductive than molybdenum (Mo) atoms. The bandgaps of monolayer MoSe<sub>2</sub> and MoTe<sub>2</sub> are 1.55 eV, 1.1 eV, respectively, extending the spectral range of TMDs from the visible to near-infrared region (WU *et al.*, 2020).

The mass production of TMDs is required for their practical applications. Currently, there are two categories of TMDs synthesis methods: bottom-up and top-down approaches. Chemical vapor deposition (CVD) and metal-organic chemical vapor deposition (MOCVD), both of which are bottom-up approaches, can synthesize wafer-scale monolayer  $MoX_2$  films. Although continuous efforts to produce large-scale wafers show promise for practical applications (YU et al., 2017; HU et al., 2023; XU et al., 2021), the cost remains high for industrial application at this stage. The mechanical exfoliation approach, a top-down approach, can produce high-quality MoX<sub>2</sub>, which is suitable for advanced fundamental research. However, its efficiency is relatively low. Another top-down approach is the liquid-phase exfoliation (LPE). LPE methods include shear exfoliation, ultrasonication exfoliation, and microfluidization exfoliation (SETHULEKSHMI et al., 2024). Sonication-assisted liquid exfoliation is the most common technique for MoX<sub>2</sub> synthesis. For example, COLEMAN et al. (2011) demonstrated the feasibility of ultrasonic-assisted liquid-phase exfoliation as early as 2011. Their findings were further supported and expanded upon in subsequent studies (KHAN et al., 2011; 2012; O'NEILL et al., 2011; BARWICH et al., 2013; COLEMAN et al., 2013; HANLON et al., 2015; GHO-LAMVAND et al., 2016; BACKES et al., 2017; HARVEY et al., 2017; SYNNATSCHKE et al., 2019; GRIFFIN et al., 2020). Though the sonication-assisted liquid exfoliation process can cause problems such as high defect rate, low stability and impaired electronic properties of the nanosheets, its advantages are: (1) simplicity, universality, and low cost, making it suitable for mass production (AKEREDOLU et al., 2024); (2) mild operating conditions (room temperature and pressure) (AGGARWAL et al., 2024), and the properties of the nanosheets being controllable by adjusting process parameters (SETHULEKSHMI et al., 2024).

The common solvents used in sonication-assisted liquid exfoliation for  $MoX_2$  synthesis are organic polymer, typically N-methyl-2-pyrrolidine (NMP) (O'NEILL *et al.*, 2012). However, the polymer is toxic and hard to remove due to its generally high boiling point. Similarly, although alternative surfactant can exfoliate  $MoS_2$  by expanding the layers, the surfactant molecules are usually difficult to recycle (MA *et al.*, 2018; POZZATI *et al.*, 2024). Recently, significant efforts have been devoted to utilizing green solvents that achieve comparable concentrations and sizes of TMDs dispersion as NMP and surfactant-based solvents. It has been demonstrated that phyto-extracted green solvents facilitate the production of few layer  $MoS_2$ 

which enhances the photo-conversion efficiency of dvesensitized solar cells and exhibits an excellent redox activity with high specific capacitance (KUMAR et al., 2023). Polarclean, Iris and Cyrene have been reported as the most promising green solvents for the production of graphene,  $MoS_2$  and  $WS_2$ . In particular, Polarclean has been highlighted due to its low defect density (OCCHIUZZI et al., 2023). RAFI et al. (2024) produced bilayered and trilayered MoS<sub>2</sub> nanosheets by employing isopropyl alcohol and deionized water in a 7:3 ratio as a cosolvent. Green solvents biomaterials are beyond the scope of this review, with relevant work summarized in other reviews (SETHULEKSHMI et al., 2024). Among organic solvents, ethanol is considered more environmentally favorable based on environmental impact, health, and safety (EHS) statements (CAPELLO et al., 2007; SHELDON et al., 2019). Hence, our review focuses on water and ethanol solvents.

To our knowledge, no review has been reported on the sonication-assisted liquid exfoliation of MoX<sub>2</sub> employing water and/or alcohol, specifically in terms of factors of process, although reviews on water-mediated exfoliation of MoS<sub>2</sub> have been reported (AGGARWAL *et al.*, 2024).

Our review summarizes the sonication-assisted exfoliation formulation using water and/or ethanol from the following three aspects: solvents selection, sonication power, and sonication time, aiming to provide a guidance on exfoliating TMDs using water and/or ethanol via the sonication-assisted exfoliation method.

# 2. Sonication-assisted exfoliation recipe

A typical sonication-assisted exfoliation process is as follows (Fig. 1). Firstly,  $MoX_2$  powder is mixed with appropriate solvents. Then, the mixture is ultrasonicated in ultrasonic instrument. Various techniques are used to prevent excessive temperature rise during sonication. For instance, intermittent ultrasound, for example, 40 seconds ultrasonic time followed by 20-second break time, are utilized. Additionally, an ice bath or water-cooling temperature control system is used to maintain a constant temperature. The resulting dispersion subsequently is centrifuged, and the

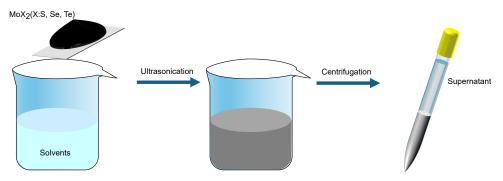


Fig. 1. Schematic diagram of the sonication-assisted exfoliation process.

supernatant is extracted. The speed and number of centrifugation steps help to roughly separate the  $MoX_2$  flakes by size.

The mechanism underlying the sonication-assisted exfoliation process has been reported (GUPTA et al., 2016; COLEMAN et al., 2011; SMITH et al., 2011; NI-COLOSI et al., 2013; LI et al., 2020). The materials discussed in detail are graphite, and the LPE solvents used are various organic solvents, such as IPA/H<sub>2</sub>O mixture, sodium dodecylbenzenesulfonate  $(SDBS)/H_2O$  mixture and NMP (LI *et al.*, 2020). Actually, LPE involves two simultaneous structural modifications: exfoliation (reduction in thickness), and fragmentation (reduction in lateral dimension). The research explains exfoliation and fragmentation processes in detail. It was found that fragmentation and exfoliation take place during LPE in three distinct stages, with the kink-band-induced peeling process being one key stages (shown in Fig. 2). In the first stage, graphite flake rupture along existing defects, and kink bands are formed due to surface acoustic waves. The second stage involves the kink bands leading to increase in chemical activity, which promotes fragmentation and exfoliation, leading to the peeling off thin graphite stripes. Then, the last stage involves the peeled graphite strips being exfoliated into thin flakes, with a minimum of ~30 layers. Although the research did not discuss exfoliation of  $MoX_2$  by LPE using water and/or ethanol, the mechanism is also applicable to the exfoliation of  $MoX_2$  by LPE using organic solvents, as both materials posses analogous 2D layered structures.

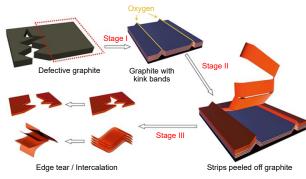


Fig. 2. Schematic diagram of the sonication-assisted LPE mechanism of graphite (reprinted with permission from (LI *et al.*, 2020)).

In general, the abovementioned procedures all have influence on the concentration and size of the exfoliated  $MoX_2$  flakes. Here, we focus on three main influencing factors, including solvents selection, sonication power and sonication time, while other aspects are beyond the scope of this review.

# 2.1. Solvents selection

There are several theories for screening solvents, such as Hansen solubility parameters (HSP), Young's equation, and Shen's method for probing and matching surface tension components (MA *et al.*, 2020). The HSP theory is commonly used, with the HSP distance  $R_a$  employed to evaluate the level of dissolution process between solvents and solutes, as described by the following equation:

$$R_a = [4(\delta_{D,\text{solv}} - \delta_{D,\text{solu}})^2 + (\delta_{P,\text{solv}} - \delta_{P,\text{solu}}) + (\delta_{H,\text{solv}} - \delta_{H,\text{solu}})^2], \qquad (1)$$

where  $\delta_D$ ,  $\delta_P$ ,  $\delta_H$  represent dispersive, polar, and hydrogen-bonding solubility parameters of a solvent and solutes, respectively (ZHOU *et al.*, 2011). The reference HSP parameters of MoX<sub>2</sub>, H<sub>2</sub>O, and ethanol are shown in Table 1.

In general, pure water is a poor solvent for  $MoS_2$ exfoliation. However, MA et al. (2018; 2020) demonstrated the feasibility of exfoliating  $MoS_2$  using water. The authors concluded that the stability of  $MoS_2$  in an aqueous solution is due to the fragmentation of the  $MoS_2$  flakes induced by sonication. Compared to graphite,  $MoS_2$  is easier to fragment. The obtained  $MoS_2$  nanosheets have sizes ranging from 100 nm to 400 nm with a few layers (5–6 layers) or multilayers (15–20 layers) in thickness. Mesoporous sheets were also observed (shown in Fig. 3a). LI et al. (2015) reported that  $MoS_2$  can be exfoliated in pure water due to defects and enlarged interlayer spacing induced by the fabrication process. ZHAO et al. (2016) exfoliated commercial  $MoS_2$  in water using a specially designed sonication instrument with a stirring function. The slipping exfoliation was achieved by the tilted rotation of  $MoS_2$  sheets during stirring. FORSBERG *et al.* (2016) exfoliated  $MoS_2$  in water using a two-step method. First, an orbital sander was used for mechanical ex-

Table 1. HSP value for MoX<sub>2</sub>, H<sub>2</sub>O, and ethanol.

	$\delta_D$ (0.5 MPa)	$\delta_P (0.5 \text{ MPa})$	$\delta_P ~(0.5 \text{ MPa})$
MoS <sub>2</sub> (Zhou <i>et al.</i> , 2011)	17 - 19	6–12	4.5 - 8.5
MoSe <sub>2</sub> (MAO <i>et al.</i> , 2018)	15.3 - 18.4	9–18	3.3 - 11.3
MoTe <sub>2</sub> (Cunningham <i>et al.</i> , 2012)	17.8	8	6.5
H <sub>2</sub> O (ZHOU <i>et al.</i> , 2011)	15.8	8.8	19.4
Ethanol (ZHOU et al., 2011)	18.1	17.1	16.9

\*The HSP parameters are obtained from (ZHOU *et al.*, 2011). Republished with permission from Angewandte Chemie International Edition, permission conveyed through Copyright Clearance Center, Inc.

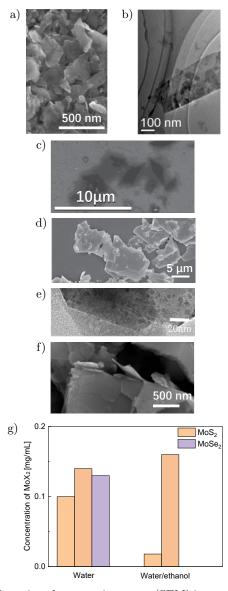


Fig. 3. Scanning electron microscope (SEM) images of exfoliated  $MoS_2$  flake (a–f) except for (e), which is the transmission electron microscope (TEM) image of exfoliated  $MoS_2$  with various vol% ethanol/water solvents. The concentration of  $MoX_2$  using water and water/ethanol solvents is shown in (g).

(a) Reprinted from (MA et al., 2018) with permission from Elsevier; (b) republished with permission from Angewandte Chemie International Edition from ZHOU et al. (2011), permission conveyed through Copyright Clearance Center, Inc; (c) republished with permission from IEEE Transactions on Nanotechnology from YUAN et al. (2021), permission conveyed through Copyright Clearance Center, Inc; (d) reprinted from WANG et al. (2013) with permission from Elsevier; (e) republished with permission from Journal of Materials Science: Materials in Electronics from YANG et al. (2017), permission conveyed through Copyright Clearance Center, Inc; (f) adapted with permission from TAGHAVI and AFZALZADEH (2021), Creative Commons License CC BY-SA 4.0.

foliation of  $MoS_2$ . Then, the obtained  $MoS_2$  powder wasexfoliated in water by sonication.  $MoS_2$  was also exfoliated in water via sonication under an  $Ar/H_2$  ato-

mosphere (GUTIÉRREZ, HENGLEIN, 1989). LIU et al. (2018a) found that bulk MoSe<sub>2</sub> can be directly exfoliated in hot water at 50 °C, achieving intense exfoliation kinetics while maintaining high quality. Based on simulation at atomic and molecular scales, it was proposed that the stable dispersion of  $MoSe_2$  nanosheets in water is achieved owing to the presence of platelet surface charges originating from edge functionalization and intrinsic polarity. A large number of atomically thin MoSe<sub>2</sub> layers are produced by 100 W sonication for 24 h and 8000 rpm centrifugation for 40 min. The lateral dimensions of the obtained MoSe<sub>2</sub> nanosheet range from 50 nm to 500 nm. A large proportion (>70%) of these layers are less than 2.0 nm thick, and >40 % of them are thinner than 1.0 nm, corresponding to monolayers. Other studies have reported that atomically thin  $MoSe_2$  platelets can be exfoliated from bulk MoSe<sub>2</sub> by 20 W sonication for 60 h and dispersed in pure water by centrifugation for 30 min under temperature control (KIM et al., 2015). The exfoliated flakes have dimensions of 200 nm to 300 nm with 2-3 layers. The concentration is shown in Fig. 3g, and lateral size and number of layers of exfoliated MoX<sub>2</sub> using water as the solvent are shown in Fig. 4, with data coming from the above-mentioned studies.

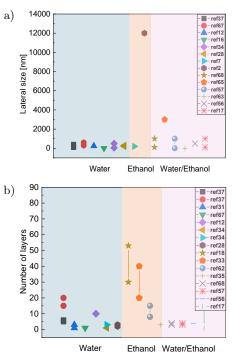


Fig. 4. MoX<sub>2</sub> using water, ethanol and water/ethanol solvents: a) lateral size; b) number of layers.

Anhydrous ethanol is used as the initial solvent for exfoliating  $MoSe_2$  and  $MoTe_2$ .  $MoSe_2$  nansosheets were obtained in anhydrous ethanol through the ultrasonicassisted LPE method and were subsequently used as a gas sensor as the ethanol solvent evaporates (CHEN *et al.*, 2019). Absolute alcohol has also been reported

to be used in the preparation of MoTe<sub>2</sub> nanoflakes (HAN et al., 2023; LIANG et al., 2020). Exfoliation was obtained by sonication for 20 h followed by centrifugation. At the end of the process, the layer thickness of the stripped nanosheet ranged from 30 to 53 atomic layers (HAN et al., 2023). Similarly, YAN et al. (2018) mixed bulk MoTe<sub>2</sub> powders with anhydrous alcohol and sonicated powders for 12 h. After centrifugation and additional processing, the number of layers of the resulting  $MoTe_2$  ranged from 8 to 15. AHMAD et al. (2021) reported MoTe<sub>2</sub> nanosheets with a lateral size of about 12  $\mu$ m by sonicating the mixture of MoTe<sub>2</sub> powder and absolute ethanol for 16 h. MoTe<sub>2</sub> nanosheets were also prepared with an ethanol-assisted ultrasound-assisted liquid-phase exfoliation (UALPE) method at 20 °C (LIU et al., 2018b). The concentration is shown in Fig. 3g, and lateral size and number of layers of exfoliated MoX<sub>2</sub> using ethanol solvent are shown in Fig. 4.

Besides single-component solvents, the HSP theory can also be used for solvent mixtures. The HSP parameters of a mixture are a linear combination of the corresponding parameters of each component, as follows:

$$\delta_{\text{blend}} = \sum \phi_{n,\text{comp}} \delta_{n,\text{comp}}, \qquad (2)$$

where  $\delta_{\text{blend}}$ ,  $\phi_{n,\text{comp}}$ ,  $\delta_{n,\text{comp}}$  represent the HSP parameters of the blend, the volume fraction of each component, the and HSP parameters of each component, respectively (ZHOU et al., 2011). By choosing water and/or alcohol with the appropriate composition, a high dispersion concentration of  $MoS_2$  can be achieved. Experimental results and theoretical predictions are consistent in showing that a 45 vol% ethanol/water provides the highest dispersion concentration, with a value of  $0.018 \pm 0.003 \text{ mg/mL}$ , which is approximately 13 times higher than that in pure ethanol and 68 times higher than that in pure water (ZHOU et al., 2011). The size of the sheets varies from 100 nm to several micrometers and their thickness is 3–4 layers (shown in Fig. 3b). Other works also report using a 45 vol% alcohol/water mixture as the solvent for  $MoS_2$  flake preparation. YUAN et al. (2021) prepared MoS<sub>2</sub> nanosheets by liquid phase exfoliation (LPE) for a formic acid gas sensor using a 45 vol% alcohol/water mixture as the solvent. The size of the nanosheets obtained is about  $3 \ \mu m$  (shown in Fig. 3c). WANG et al. (2013) also prepared MoS<sub>2</sub> nanosheets by dispersing  $MoS_2$  powder in a 45 vol% ethanol/water mixture, with a thickness of 3–4 layers and the size of nanosheets ranging from tens of nanometers to several micrometers (shown in Fig. 3d). HUANG et al. (2024) prepared few-layer MoS<sub>2</sub> flakes with an average thickness of 7 nm by sonication in  $45 \ \mathrm{vol}\%$  ethanol/water mixture at  $240 \ \mathrm{W}$  for  $90 \ \mathrm{min}.$ In addition to the 45 vol% ethanol/water mixture, other proportions of ethanol/water have also been reported for liquid-phase exfoliation of  $MoS_2$  flakes.

For example,  $MoS_2$  quantum dots were obtained by dispersing defected  $MoS_2$  nanosheets into a 25 vol% ethanol/water solution. Due to the inherent defects in  $MoS_2$ , the average lateral size of acquired  $MoS_2$ quantum dots is 3.6 nm – shown in Fig. 3e (YANG et al., 2017). A 23 vol% ethanol/deionized water solution was also reported to be utilized to exfoliate  $MoS_2$ , yielding flakes with an average of 4 layers and a lateral size of 500 nm - shown in Fig. 3f (TAGHAVI et al., 2021). Furthermore, a 50 vol% ethanol/water solvent mixture has been reported to exfoliate  $MoS_2$ , resulting in nanosheets with lateral sizes of several micrometers and damaged surface edges (PRABUKUMAR et al., 2018; JIN et al., 2020). Compared to the same proportion of NMP/water, this exfoliation efficiency is poor (PRABUKUMAR et al., 2018). HALIM et al. (2013) used Young's equation to determine the liquid-solid interfacial energy and predicted that the optimal cosolvent of alcohol-water mixtures should have a surface tension between  $30 \text{ mJ/m}^2$  and  $35 \text{ mJ/m}^2$ . The concentration lateral size and number of layers of exfoliated MoX<sub>2</sub> using water/ethanol solvent are shown in Figs. 3g and 4, respectively.

#### 2.2. Sonication power

In addition to the solvent type, which affects the quality of the final MoX<sub>2</sub> production in ultrasoundassistant liquid exfoliation, the ultrasonic power also plays a crucial role. Sonication power is an important parameter influencing the exfoliation process. The size of exfoliated  $MoS_2$  flakes increases as the sonication power increases from 38.5 W, 47 W, to 65.5 W with bath sonication. At 84 W, the  $MoS_2$  flakes begin to agglomerate (TAGHAVI, AFZALZADEH, 2021). This phenomenon can be explained by the collapse of the highenergy bubbles, which increases the size and number of bubbles. As a result, the shock waves produced by sonication are reduced while the bubble implosions increase. Unlike bath sonication, probe sonication uses an ultrasound probe to transmit vibrations. HAU et al. (2021) synthesized MoS<sub>2</sub> for 8 h via probe sonication at 420 W using a water/ethanol with a volume ratio of 2:1.

Some divergence exists between sonication-assisted LPE using water and/or ethanol and organic solvents. For instance, using a mixture of chloroform and acetonitrile in a 65:35 ratio as solvents for LPE, the average size of MoS<sub>2</sub> nanosheets decreases as the ultrasonic power increases from 350 W, 450 W to 550 W. Meanwhile, the concentration of produced MoS<sub>2</sub> increases correspondingly. This phenomenon is explained by the cavitation effect and micro-jet effect induced by ultrasound. The cavitation effect is the primary force for exfoliating layered MoS<sub>2</sub>, involving the process of the formation, growth, and implosive collapse of bubbles. Simultaneously, the micro-jet effect induced by the collapse of bubbles is the force that fragments the

 $MoS_2$  sheets (ZHANG *et al.*, 2014). The effect of ultrasonic power on exfoliation has also been studied using NMP as a solvent (QIAO *et al.*, 2014). The ultrasonic power was controlled from 100 W, 200 W, 250 W, 285 W, 320 W, 350 W to 400 W. The concentration of nanosheets increased as the sonication power increased, and then decreased after 320 W. Meanwhile, the size of the nanosheets initially decreased and then increased after 320 W. This behavior is associated with the ultrasonic cavitation effect. At low input power, the covalently bonded S-Mo-S sheet are broken into small flakes due to inertial cavitation. However, at high input power, the breaking intensity decreases due to fewer large bubbles being generated, a phenomenon known as ultrasonic cavitation shielding effect.

### 2.3. Sonication time

Sonication time is another important parameter. TAGHAVI and AFZALZADEH (2021) systematically studied the effect of sonication time on the exfoliation of  $MoS_2$  using a mixture of 77 % deionized (DI) water and 23 vol% ethanol by volume. They found that the size of MoS<sub>2</sub> flakes increases as the effective sonication time increases from 15 min to 60 min, but then decreases with prolonged sonication time. This is due to the agglomeration process. A similar effect has been observed in the LPE of  $MoS_2$  using NMP (O'NEILL et al., 2012). The dimensions of  $MoS_2$  flakes increase after 23 h of sonication, reaching a maximum after 60 h, and then decrease after 60 h of sonication. MITTAL et al. (2023) reported that the number of exfoliated MoSe<sub>2</sub> layers in DI water and ethanol decreases as the sonication time increases from 10 min to 60 min. LIU et al. (2018a) studied the effect of sonication time on exfoliation of MoSe<sub>2</sub> using water at 50 °C. For comparison, the authors sonicated bulk MoSe<sub>2</sub> for 8 h and 24 h, and found that the layers after 8 h of sonication could notwithstand higher centrifugal speed. However, bulk MoSe<sub>2</sub> could be broken into high-quality layers at a longer ultrasound time (24 h) due to more sufficient exfoliation.

XU et al. (2024) first compared the effects of different ethanol contents on the dispersibility of  $MoTe_2$ , and then analyzed the relationship between sonication time and the thickness of nanosheets at intervals of 0.5 h, 1.5 h, 2.5 h, 3.5 h, 4.5 h, and 5.5 h. The results showed that the average thickness of the nanosheets decreased as the sonication time increased.

## 3. Application, perspective and conclusions

Exfoliated TMDs using water and/or ethanol solvents enable a wide range of applications, such as electrochemical application as supercapacitor electrodes, photoelectrochemical applications for photocurrent response material (KAJBAFVALA *et al.*, 2018), mechani-

cal reinforcement in polymers (O'NEILL et al., 2012). electrocatalysts for hydrogen evolution reactions, hazardous gas sensor, batteries, surface coatings, and more. Due to its high carrier mobility, strong spinorbit coupling, and extensive light absorption, MoSe<sub>2</sub> is considered as one of the most promising materials for optoelectronics in TMDs, making it suitable for flexible, lightweight optoelectronic devices (PATEL et al., 2019).  $MoSe_2$  exfoliated by alcohol solvents can also be applied to gas sensors by taking advantage of the volatilization of alcohol (ZHOU et al., 2011). MoTe<sub>2</sub> can be transformed into many types of lasers following specific processing. Additionally, sonicationassisted LPE using water and/or ethanol solvents is an environment-friendly, low-cost, and easy-to-operate method for scaling up mass production of TMD flakes, making it suitable for industrial practical applications (CIESIELSKI, SAMORÌ, 2014). Non-toxic, environmentally friendly solvents and dispersants can extend the range of 2D TMD inks (LEE et al., 2020). From a technological perspective, sonication-assisted LPE using water and ethanol not only facilitates upscaled production of TMDs flakes comparable to organic solvents, but it is also an economical and practical solution, as water and ethanol do not require additional postprocessing for environmental compliance.

In this review, the preparation of MoX<sub>2</sub> flakes using the sonication-assisted LPE method with water and/or ethanol was summarized. Although many parameters influence this method, the review focused on three main parameters: solvent selection, sonication power, and sonication time. Solvent selection refers to the ratio of water and/or alcohol used. Related studies were summarized, revealing that a 45 vol% alcohol/water mixture is the optimal solvent for  $MoS_2$ , as explained by HSP theory. The effects of sonication power exhibit some inconsistencies, and even some divergences exit between LPE using water and/or etahnol solvents versus organic solvents. This variation may be attributed to differences in sonication equipment used by various research groups. Regarding sonication time, the size of  $MoS_2$  flakes initially increases, and then decreases as sonication time increases. This phenomenon is analogous to that observed when using organic solvents. To further analyze the mechanism behind the LPE method using water and/or ethanol solvents, the advanced LPE mechanism, which includes three stages, is summarized from the literature. Finally, the wide applications of exfoliated MoX<sub>2</sub> flakes and the future outlook for the LPE method using water and ethanol solvents were discussed.

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