#### **ORIGINAL RESEARCH ARTICLE**



# A Thrifty Liquid-Phase Exfoliation (LPE) of MoSe<sub>2</sub> and WSe<sub>2</sub> Nanosheets as Channel Materials for FET Application

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Received: 11 September 2022 / Accepted: 11 January 2023 / Published online: 6 February 2023 © The Minerals, Metals & Materials Society 2023

# Abstract

Two-dimensional materials are trending nowadays because of their atomic thickness, layer-dependent properties, and their fascinating application in the semiconducting industry. In this work, we have synthesized  $MoSe_2$  and  $WSe_2$  nanosheets (NSs) via a liquid-phase exfoliation method and investigated these NSs as channel materials in field-effect transistors (FET). The x-ray diffraction (XRD) pattern revealed that the synthesized NSs have a 2H phase with 0.65 nm *d*-spacing which belongs to the (002) Miller plane. Transmission electron microscopy (TEM) studies revealed that  $MoSe_2$  and  $WSe_2$  have a nanosheet-like structure, and the average lateral dimensions of these NSs are ~25 nm and ~63 nm, respectively. From Raman spectra, we found that the intensity of the  $A_{1g}$  vibrational mode decreases with the reduction in the number of layers. UV-visible spectroscopy revealed that the bandgap values of  $MoSe_2$  and  $WSe_2$  NSs are 1.55 eV and 1.50 eV, respectively, calculated using the Tauc equation. The output and transfer characteristics of the FET devices reveals that the fabricated FETs have good ohmic contact with the channel material and an ON/OFF current ratio of about 10<sup>2</sup> for both devices. This approach for the fabrication of FET devices can be achieved even without sophisticated fabrication facilities, and they can be applied as gas sensors and phototransistors, among other applications.

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#### **Graphical Abstract**



Keywords  $MoSe_2 \cdot WSe_2 \cdot nanosheets \cdot LPE \cdot FET$ 

# Introduction

The channel length of a transistor for electronic applications has been continuously reduced over the last several decades owing to increased demand and reduced cost, with no compromise of performance, as predicted by Moore back in 1965. The miniaturization of devices is a major challenge for the silicon industry. To satisfy the ever-growing demand of electronic and optoelectronic applications, many other low-dimensional materials have been explored such as carbon nanotubes (CNTs),<sup>1,2</sup> graphene,<sup>3,4</sup> transition metal dichalcogenides (TMDs),<sup>5–7</sup> and MXenes.<sup>8,9</sup> Among these materials, semiconducting TMDs are the first descendants of two-dimensional (2D) materials which are continuously explored for nano-electronic applications.

TMDs have  $MX_2$  type hexagonal structure, where M is a transition metal and X (S, Se, Te) is a chalcogen atom.<sup>10,11</sup> In TMDs, there is strong covalent bonding in the hexagonal layer between the transition metal atom (M) and chalcogen atom (X) and weak van der Waals (vdW) bonding between the adjacent layers. Due to the vdW interaction, TMDs can

be easily peeled off into monolayers having atomic thickness. These atomically thin structures have layer-dependent properties which offer an advantage over conventional materials, such as direct-bandgap transition with a decreased in number of layers and high electrical conductivity.<sup>12,13</sup> TMDs can be synthesized by various methods including micromechanical cleavage,<sup>14,15</sup> chemical vapor deposition (CVD),<sup>16</sup> and the hydrothermal/solvothermal method,<sup>17-20</sup> whereas in the liquid-phase exfoliation (LPE) method,<sup>21,22</sup> the ultrasonication process emerges as a powerful tool for the exfoliation of 2D materials. The solution thermodynamics describe the mechanism of exfoliation in the LPE method. Several other TMDs have been explored as channel materials for field-effect transistors (FETs) such as HfS<sub>2</sub>, MoS<sub>2</sub>, and phosphorene,<sup>23-25</sup> which offer better FET performance including subthreshold swing, mobility, and high current ON/OFF ratio. Molybdenum selenide (MoSe<sub>2</sub>) and tungsten selenide (WSe<sub>2</sub>) also belong to the class of 2D-TMD materials and are potential candidates as channel materials in FETs. The evolution of the electronic structure in chemical vapor transport (CVT)-grown atomically thin WS<sub>2</sub> and WSe<sub>2</sub> structures was reported by Zhao et al., who confirmed a 100- and 1000-fold enhancement in photoluminescence (PL) intensity compared to bulk WS<sub>2</sub> and WSe<sub>2</sub>, respectively.<sup>26</sup> Varghese et al. demonstrated the direct transfer of exfoliated MoSe<sub>2</sub> nanosheets (NSs) with unprecedentedly large holes almost 15 µm in diameter.<sup>27</sup> A WSe<sub>2</sub> channel-based FET sensor for SARS-CoV-2 was fabricated by Hafshejani et al.<sup>28</sup> with a detection limit as low as 25 fg/µL in 0.01X phosphate-buffered saline. Jung et al. reported the fabrication of a CVDgrown MoSe<sub>2</sub> channel FET device with p-type ambipolar behavior, and further studied their application as phototransistors. They found that the field effect mobility of the device was 10 cm<sup>2</sup>/Vs, and it exhibited photo-responsivity of 93.7 A/W.<sup>29</sup> Yoo et al. synthesized an n-type MoSe<sub>2</sub> grown by CVD, and the mobility of the device was 75 cm<sup>2</sup>/Vs with  $I_{ON/OFF} \sim 10^{5.30}$  Moon et al. reported a modified resistance network to extract the transfer length and contact resistivity of WSe<sub>2</sub> channel-based FET devices. For *n*-type and *p*-type WSe<sub>2</sub>-FET devices, the sheet resistance values were 38 k $\Omega$ / sq and 28 k $\Omega$ /sq, respectively.<sup>31</sup> The interface of the metal contact and WSe<sub>2</sub> channel FET device was studied by Liu et al., in which they observed that the In-WSe<sub>2</sub> FET showed better electrical properties, high ON current and electron mobility up to 210 µA/µm and 142 cm<sup>2</sup>/Vs, respectively.<sup>32</sup> However, the performance metrics such as ON/OFF ratio, mobility, and ON current of devices are not as high for practical application, so more study is still required in this direction to enhance the performance parameters of these devices. After an exhaustive survey of the previous reports, we found that exfoliation of the 2D materials from bulk MoSe<sub>2</sub> and WSe<sub>2</sub> in *N*-methyl-pyrrolidone (NMP) solution via the LPE method is easy and cost-effective. Thus, we synthesized MoSe<sub>2</sub> and WSe<sub>2</sub> NSs using the LPE method. These exfoliated NSs were characterized by x-ray diffraction (XRD), field emission scanning electron microscopy

(FESEM), transmission electron microscopy (TEM) and Raman spectroscopy to study the morphology and structure of the synthesized NSs. Ultraviolet–visible spectroscopy was used to study the optical properties of NSs. Further, these NSs were transferred on a commercially available pre-patterned organic FET (OFET) device for the investigation of the electrical properties. Because of the atomic-scale thickness, 2D material-based device fabrication is very difficult; most researchers fabricate such devices using electron beam lithography (EBL), which requires highly complex systems that are too costly and thus not practical. Our technique provides a simple approach for the fabrication of 2D materialbased FET devices and opens new vistas for other researchers to work in the field of nano-electronics.

# **Experimental Section**

MoSe<sub>2</sub> and WSe<sub>2</sub> bulk powder (Alfa Aesar,  $90\% < 2 \mu m$ ) and NMP (Sigma Aldrich) were used to synthesize the 2D-MoSe<sub>2</sub> and WSe<sub>2</sub> NSs by the LPE synthesis route because of their high yield and cost-effectiveness. In this process, we started with 10 mg/ml concentration of bulk powder in NMP in a glass vial. The prepared suspension was sonicated for 8 h in a bath sonicator. Once the sonication process was complete, the vials were left undisturbed overnight and solutions were centrifuged at 3000 rpm for 15 min. The supernatant was collected in glass vials. The supernatants were then investigated via various characterization techniques. The exfoliation of MoSe<sub>2</sub> and WSe<sub>2</sub> NSs can be understood by the schematic shown in Fig. 1. Hygroscopic



Fig. 1 Schematic of synthesis process of MoSe<sub>2</sub> and WSe<sub>2</sub> nanosheets.

and lactams have rich chemistry involving nucleophilic addition, elimination and ring interaction.<sup>33</sup> For such processes, sonication is complemented by the presence of O<sub>2</sub> and H<sub>2</sub>O from atmosphere. Thus, the extremely active intermediate species such as radicals or hyperoxides (in the case of NMP, N-methylsuccinimide [NMS] is created by auto-oxidation of NMP) promote the exfoliation. Some reports also suggest that auto-oxidation is triggered in the absence of sonication. It is supposed that the MX<sub>2</sub> in NMP undergoes edge oxidation and partial dissolution which releases the highly charged anionic clusters. As a consequence, the negatively charged defects and anionic clusters are adsorbed on the surface of MX<sub>2</sub>, leading to the electrostatic repulsion of MX<sub>2</sub> NSs in the colloids.<sup>34,35</sup> Further, these 2D NSs were drop-cast on OFET chips (OFET Gen4 devices, Fraunhofer, Germany) to study their behavior as channel material. The source/drain is composed of gold with a thickness of 40 nm, separated by a 230-nm-thick dielectric layer of silicon dioxide (SiO<sub>2</sub>). Structural and morphological studies of the samples were carried out by 200 keV, high-resolution transmission electron microscopy (HR-TEM; JEOL JEM-F200 with a Gatan OneView camera), FESEM (JEOL, JSM-7610F), XRD (PANalytical Empyrean with Cu K $\alpha$  as source  $\lambda = 1.54$  Å), and Raman spectroscopy (Horiba Jobin-Yvon Labram HR system), respectively. The optical properties of the prepared samples were studied by UV-visible spectroscopy (UV-2401PC UV-visible spectrometer, Shimadzu). A four-probe setup (Keithley 2612A SourceMeter ) was used to measure the electrical properties of the FET device.



Fig. 2 XRD pattern of  $MoSe_2$  and  $WSe_2$  nanosheet peaks at 13.45° and 13.55°, respectively, confirms the 2H phase.

### **Results and Discussion**

#### **XRD Analysis**

The XRD patterns of  $MoSe_2$  and  $WSe_2$  NSs are shown in Fig. 2. The peaks observed at 13.45° and 13.55° belong to the (002) Miller planes for  $MoSe_2$  and  $WSe_2$  NSs, respectively (JCPDS 98-004-9800 and 98-004-0752).<sup>29,36,37</sup> The peak corresponding to 24° for both  $MoSe_2$  and  $WSe_2$ belongs to quartz. The peaks at 13.45° and 13.55° for  $MoSe_2$  and  $WSe_2$  NSs confirm that the synthesized NSs have a hexagonal 2H structure with space group P6<sub>3</sub>/mmc which is semiconducting in nature.<sup>36</sup> The broadness of (002) plane indicates the formation of a few-layer-thick layered material.<sup>38</sup> The interplanar (*d*) spacing for the NSs is calculated for (002) plane using Bragg's law:<sup>39</sup>

$$2d\sin\theta = n\lambda\tag{1}$$

where d,  $\lambda$  and  $\theta$  are the *d*-spacing, x-ray wavelength, and Bragg's angle, respectively. The *d*-spacing for MoSe<sub>2</sub> and WSe<sub>2</sub> is 0.65 nm as calculated for the (002) plane, which is in good agreement with the values from the literature. The micro-strain values for the NSs were calculated using the following equation:<sup>40</sup>

$$\epsilon = \frac{\beta * \cot \theta}{4} \tag{2}$$

where  $\epsilon$  is the root mean square value of the micro-strain,  $\beta$  is the full width at half maximum (FWHM) and  $\theta$  is the Bragg angle. The strain for MoSe<sub>2</sub> and WSe<sub>2</sub> is 4.22 and 1.62, respectively. The defect state created during the exfoliation process is responsible for the micro-strain produced in the MoSe<sub>2</sub> and WSe<sub>2</sub> structure, explained in the experimental section.

# **TEM Analysis**

The morphology and internal structure of MoSe<sub>2</sub> and WSe<sub>2</sub> NSs were studied by TEM. For TEM measurement, suspension of MoSe<sub>2</sub> and WSe<sub>2</sub> NSs was drop-cast onto a carbon-coated copper grid. The TEM and selected area electron diffraction (SAED) patterns of MoSe<sub>2</sub> and WSe<sub>2</sub> NSs are shown in Figs. 3 and 4. The average lateral dimension of MoSe<sub>2</sub> NSs is ~25 nm, which is shown in the inset of Fig. 3a. The zoomed image of the MoSe<sub>2</sub> NSs is shown in Fig. 3b, and the *d*-spacing is calculated as 0.67 nm, 0.24 nm, and 0.28 nm, corresponding to the (002), (103), and (100) Miller planes, respectively.<sup>41–43</sup> The TEM image of WSe<sub>2</sub> NSs is shown in Fig. 5a, and the inset shows the image of the NS with interplanar spacing of 0.62 nm belonging to the (002) Miller plane, which matches with the XRD result.<sup>44</sup> The average lateral dimension of WSe<sub>2</sub> NSs is~63 nm. The SAED pattern for MoSe<sub>2</sub> and WSe<sub>2</sub> is shown in Figs. 3b and 5b, respectively, and the circular diffraction



Fig. 3 (a) TEM image and inset showing the HR-TEM image and (b) SAED pattern of  $MoSe_2 NSs$ .



Fig. 4 EDX spectra of MoSe<sub>2</sub> NSs exfoliated in NMP.



Fig. 5 (a) TEM image and inset showing the HR-TEM and (b) SAED pattern of  $WSe_2 NSs$ .



Fig. 6 EDX spectra of WSe<sub>2</sub> NSs exfoliated in NMP.

pattern indicates that the NSs are polycrystalline in nature. The SAED pattern from multiple smaller grains may be responsible for the ring-like pattern, which was similarly reported for WS<sub>2</sub> by Shinde et al.<sup>45</sup> The energy-dispersive X-ray (EDX) spectra

for  $MoSe_2$  and  $WSe_2$  are shown in Figs. 4 and 6, respectively, suggesting that there is no impurity present in the synthesized NS dispersion.

#### **Raman Analysis**

Raman investigation of the  $MoSe_2$  and  $WSe_2 NSs$  was carried out at 488 nm laser excitation, and three peaks observed in the range of 100–400 cm<sup>-1</sup> were identified. The phonon modes were assigned by comparing the peaks with the existing literature. The 2H symmetry of  $MoSe_2$  and  $WSe_2$  belongs to the space group  $D_{6h}^4$ , and there are 12 modes of lattice vibration at the center of the Brillouin zone:<sup>46–48</sup>

$$A_{1g} + 2A_{2u} + B_{1u} + 2B_{2g} + E_{1g} + 2E_{1u} + E_{2u} + 2E_{2g}$$
(3)

where  $A_{1u}^1$  and  $E_{1u}^1$  are the acoustic mode,  $A_{1u}^2$ ,  $E_{1u}^2$  are the infrared-active modes, and  $A_{1g}$ ,  $E_{1g}$ ,  $E_{2g}^1$ ,  $E_{2g}^2$  are the Ramanactive modes. These modes are divided into six pairs, where one is symmetric and the other is antisymmetric with respect



Fig. 7 Raman spectra of MoSe<sub>2</sub> and WSe<sub>2</sub> nanosheets.

to the inversion around the center lying around the Se-atom on both sides of the interlayer gap. The Raman spectra of MoSe<sub>2</sub> and WSe<sub>2</sub> NSs are shown in Fig. 7. The black line represents spectra of MoSe<sub>2</sub>, and the red line belongs to WSe<sub>2</sub>. The peaks observed at 169 cm<sup>-1</sup>, 241 cm<sup>-1</sup>, and 286 cm<sup>-1</sup> are first-order Raman peaks assigned to phonon modes of MoSe<sub>2</sub> corresponding to  $E_{1g}$ ,  $A_{1g}$ , and  $E_{2g}^1$ . respectively, which match with the literature.<sup>46</sup> The bulk  $A_{1g}$  is reported at 243 cm<sup>-1</sup> in the literature, which is shifted to 2 cm<sup>-1</sup> towards the lower wavenumber due to the decrease in interplanar restoring force.<sup>49</sup> This also confirms the exfoliation of bulk MoSe2. Prominent Raman modes for WSe2 NSs were observed at 175 cm<sup>-1</sup>, 251 cm<sup>-1</sup>, and 282 cm<sup>-1</sup> belonging to the  $E_{1g}$ ,  $A_{1g}$  and  $E_{2g}^1$ , respectively.<sup>48,50</sup> Similarly, the  $A_{1g}$  vibrational frequency for bulk WSe<sub>2</sub> is reported at  $253 \text{ cm}^{-1}$ ,<sup>48</sup> which is shifted by 2 cm<sup>-1</sup> toward the lower wavenumber. The Raman spectra of MoS<sub>2</sub> has been studied extensively to identify the thickness of NSs, whereas to the best of our knowledge, no such study has been reported for WSe<sub>2</sub> and MoSe<sub>2</sub> NSs that are thinner by a few layers.

## **UV–Visible Spectroscopy**

It is well known that the dimensionality affects the optical properties of materials. Therefore, absorption spectroscopy is used to study the optical properties of the synthesized NSs. The absorbance spectra of  $MoSe_2$  and  $WSe_2$  NSs are shown in Fig. 8a, and the zoomed-in image shows the absorbance peak of NSs. A broad peak in the region of 400–500 nm for both  $MoSe_2$  and  $WSe_2$  NSs can be attributed to the direct transition at the M-point of the Brillouin zone or due to the



Fig. 8 (a) Absorbance spectra of  $MoSe_2$  and  $WSe_2$  nanosheets, where the inset depicts the absorption peaks at 741 and 748 nm for  $MoSe_2$  and  $WSe_2$ , respectively, (b) Tauc plots of  $MoSe_2$  and  $WSe_2$  nanosheets.



Fig. 9 (a, b) FESEM image of  $MoSe_2$  and  $WSe_2$  nanosheet channel-based FET devices.

Van Hove singularity. The peaks at 741 nm and 748 nm for  $MoSe_2$  and  $WSe_2$  NSs, respectively, correspond to the direct excitonic transition at the K-point in the Brillouin zone. The direct bandgap of the NSs is calculated by using the Tauc equation given below:<sup>51,52</sup>

$$(\alpha h\nu)^2 = A(h\nu - E_g) \tag{4}$$

where  $\alpha$ ,  $h\nu$ ,  $E_g$ , and A are the absorption coefficient, photo energy, bandgap, and constant, respectively. The bandgap energy of MoSe<sub>2</sub> and WSe<sub>2</sub> NSs is calculated by extrapolating the linear part of the Tauc plot  $[(\alpha h\nu)^2 Vsh\nu]$ . The Tauc plot is shown in Fig. 8b. The bandgap values of MoSe<sub>2</sub> and WSe<sub>2</sub> NSs are 1.55 eV and 1.50 eV, respectively, which is higher than their bulk counterpart as reported in the literature.<sup>53,54</sup> This difference in bandgap suggests that the bulk MoSe<sub>2</sub> and WSe<sub>2</sub> are reduced to few-layer-thick NSs.<sup>52</sup>

### **Electrical Properties of FET Devices**

To investigate the electrical properties, the synthesized NSs were deployed as channel materials on a pre-patterned OFET substrate. The prepared NSs were drop-cast on the open-channel OFET devices with a channel length of 2.5  $\mu$ m, as shown in Fig. 9a and b. The zoomed-in image of the channel confirms that MoSe<sub>2</sub> and WSe<sub>2</sub> NSs have been deposited over the channel.

The devices were annealed at 473 K prior to electrical measurement in order to remove any residual contamination. The electrical measurement was performed on a Keithley 2612A SourceMeter in ambient atmosphere. The Au electrode acted as the source and drain electrode. The transfer and output characteristic curve of the MoSe<sub>2</sub>-FET

is shown in Fig. 10a and b. Figure 10a confirms a linear relationship between the drain current and drain voltage up to -0.5 V (threshold voltage), indicating good ohmic contact between the Au electrode and MoSe<sub>2</sub> channel material. In output characteristics, the  $V_{gs}$  varies from 0 V to 5 V. The drain current decreases with the application of gate voltage  $(V_{gs})$  in the MoSe<sub>2</sub> channel FET device. This decrease in drain current (increase in resistance) suggest that the depletion width increases for the MoSe<sub>2</sub>-FET device. When the applied voltage exceeds the threshold voltage, the device turns ON, and the current that flows through the channel in this situation is referred to as the ON current. The term "OFF current" refers to the current that exists below the threshold voltage, and it changes (increases) exponentially as the drain voltage increases. When the FET is in the OFF condition, there is no channel formed between the drain and source terminals. When the FET is in the other two regions, i.e. linear and saturation regions, it is in the ON condition, and there is a channel formed between the drain and source terminals. In transfer characteristics, the maximum ON current is  $4.8 \times 10^{-9}$  A and the OFF current is  $2.37 \times 10^{-11}$  A. The ON/OFF current ratio for the devices is  $10^2$ . From the output characteristics of the WSe<sub>2</sub>-FET (shown in Fig. 10c), it is clear that there is a linear relationship between the channel material and the Au electrode. The saturation region is clearly visible in the output characteristics. The drain current increases with an increase in gate voltage, which suggests that upon application of the gate voltage there is an increase in the depletion width of the WSe<sub>2</sub> FET device. This change in depletion width is responsible for the better FET performance. The transfer characteristics of the WSe<sub>2</sub> FET device are shown in Fig. 10d. The maximum ON and OFF current for the WSe<sub>2</sub>-FET devices is  $7.65 \times 10^{-10}$  A and  $3.55 \times 10^{-12}$ A, respectively. The ON/OFF current ratio for the device



Fig. 10 (a-d) Transfer and output characteristics of MoSe<sub>2</sub> and WSe<sub>2</sub> nanosheet channel-based FET devices.

Table I MoSe <sub>2</sub> and WSe <sub>2</sub> FET   parameters derived from output and transfer characteristics	S. No.	FET device	Threshold volt- age (Vth)	ON current (A)	OFF current (A)	ON/OFF ratio
	1	MoSe <sub>2</sub> -FET	-0.5	$4.8 \times 10^{-9}$	$2.37 \times 10^{-11}$	10 <sup>2</sup>
	2	WSe <sub>2</sub> -FET	-0.5	7.65 × 10 <sup>-10</sup>	$3.55 \times 10^{-12}$	10 <sup>2</sup>

is  $10^2$ . The performance of the devices is on par matches with the reported literature.<sup>55–57</sup> The FET parameters of both devices are given in Table I. This device fabrication approach could help researchers in the study of the TMD-FET for application in different types of sensors, such as gas sensors, biosensors, humidity sensors, photodetectors, and photovoltaic applications. The performance of the fabricated TMD FETs can be further enhanced by controlling

the various processing issues including the generation of surface defects, presence of charge impurities, localized charge distribution, and trapped charges. Due to the presence of these defects, Coulomb scattering takes place, which causes high contact resistance between the metal and TMD material interface and hence overall degradation of device performance.

# Conclusion

LPE was used to synthesize MoSe<sub>2</sub> and WSe<sub>2</sub> NSs, and these 2D-NSs were used as a channel material for FET applications. MoSe<sub>2</sub> and WSe<sub>2</sub> have a few-layer-thick 2H semiconducting phase. From Raman spectra, a vibration mode  $A_{1e}$  for MoSe<sub>2</sub> and WSe<sub>2</sub> exists, which confirms the reduction in the number of layers of 2D materials. From the UV-visible spectroscopy, we found that the bandgap values of MoSe<sub>2</sub> and WSe<sub>2</sub> NSs are 1.55 eV and 1.5 eV, respectively. These exfoliated NSs were cast off as the channel material for the OFET chips. A metal-semiconductor interface was formed at the contact and exhibited Schottky diode-like behavior, which is shown in the transfer characteristics of FET devices, and both devices exhibited *n*-type behavior. The electrical properties revealed that the ON/OFF current ratio for  $MoSe_2$  and  $WSe_2$  is  $10^2$ . This approach for the fabrication of FET devices could advance the study of FET-based devices in sensing and photovoltaics.

Acknowledgments The authors from Amity Institute of Nanotechnology (OPS and RS) acknowledge the financial support received from DRDO-SSPL (CARS-1115/CARS-64/TS/SPL/18), New Delhi, under the project. The authors are thankful to IUAC for extending the FESEM facility funded by the Ministry of Earth Sciences (MoES) under the Geochronology project [MoES/P.O.(Seismic)8(09)-Geochron/2012].

Conflict of interest There are no conflicts to declare.

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