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PATENT CERTIFICATE
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पेटेंटी / Patentee : INDIAN INSTITUTE OF TECHNOLOGY MADRAS (IIT Madras)
आविष्कारक (जहां लागू हो) / Inventor(s) : 1.Thalappil Pradeep 2.Biswajit Mondal 3.Ananthu Mahendranath 4.Anirban Som 5.Sandeep Bose 6.Tripti Ahuja 7.Avula Anil Kumar 8.Jyotirmoy Ghosh

प्रमाणित किया जाता है कि पेटेंटी को उपरोक्त आवेदन में यथाप्रकटित REMOVAL OF LEAD FROM WASTE WATER USING NANOSCALE MOS2 नामक आविष्कार के लिए, पेटेंट अधिनियम, १९७० के उपबंधों के अनुसार आज तारीख 11th day of December 2017 से बीस वर्ष की अवधि के लिए पेटेंट अनुदत्त किया गया है।

It is hereby certified that a patent has been granted to the patentee for an invention entitled REMOVAL OF LEAD FROM WASTE WATER USING NANOSCALE MOS2 as disclosed in the above mentioned application for the term of 20 years from the 11th day of December 2017 in accordance with the provisions of the Patents Act,1970.



अनुदान की तारीख : 22/04/2021
Date of Grant :

पेटेंट नियंत्रक
Controller of Patent

टिप्पणी - इस पेटेंट के नवीकरण के लिए फीस, यदि इसे बनाए रखा जाना है, 11th day of December 2019 को और उसके पश्चात प्रत्येक वर्ष में उसी दिन देय होगी।

Note. - The fees for renewal of this patent, if it is to be maintained will fall / has fallen due on 11th day of December 2019 and on the same day in every year thereafter.

FORM 2
THE PATENTS ACT, 1970
(39 OF 1970)
&
The Patents Rules, 2003
COMPLETESPECIFICATION
(Refer section 10 and rule 13)

TITLE OF THE INVENTION:

REMOVAL OF LEAD FROM WASTE WATER USING NANOSCALE MoS₂

2. APPLICANT:

(A) NAME: **INDIAN INSTITUTE OF TECHNOLOGY MADRAS (IIT Madras)**

(B) NATIONALITY: Indian

(C) ADDRESS: **INDIAN INSTITUTE OF TECHNOLOGY MADRAS**

IIT P.O

Chennai - 600 036

3. Preamble to the Description

COMPLETESPECIFICATION

The following specification particularly describes the invention and the manner in which it has to be performed.

COMPLETE SPECIFICATION

TITLE OF THE INVENTION

5 **REMOVAL OF LEAD FROM WASTE WATER USING NANOSCALE MoS₂**

FIELD OF THE INVENTION

The present invention relates to a chemical reactivity of MoS₂ nanosheets with Pb²⁺ and Pb⁴⁺ in solution forming PbMoO₄. This reactivity can be employed for removing toxic lead in
10 solutions.

BACKGROUND OF THE INVENTION

Inorganic analogues of graphene, due to their unusual electrical [Ma X et al., Adv. Mater. Res. (Durnten-Zurich, Switz.) 2013, 651, 193-197, 6 pp; Kim, T.Y et al. ACS Nano 2016, 10, 2819-2826, Kim, J. S., et al., ACS Nano 2016, 10, 7500-7506], electronic [Radisavljevic B et al., Nat Nano 2011, 6, 147-150; Wei, X.-L et al., Journal of Materials Chemistry A 2014, 2, 2101-2109; Steinhoff A et al., Nano Letters 2014, 14, 3743-3748], magnetic[Gao N et al., J. Phys. Chem. C 2017, 121, 12261-12269; Tongay S et al., Appl. Phys. Lett. 2012, 101, 123105/1-123105/4; Saab M et al., The Journal of Physical Chemistry C 2016, 120, 10691-10697] and
15 catalytic properties[Li Y et al., Journal of the American Chemical Society 2011, 133, 7296-7299; Lukowski M. A. et al., Journal of the American Chemical Society 2013, 135, 10274-10277; Voiry, D et al., Nano Letters 2013, 13, 6222-6227; Pi Y et al., ACS Sustainable Chem. Eng. 2017, 5, 5175-5182], are fascinating materials which have made great inroads into materials science of the recent past. While their novel physical properties have been fascinating, most such
20 investigations have left the nanostructures undisturbed. As the physical properties have been inherent to the nanosystems, retention of the structure has been essential for the properties. Likely chemical changes on them during the processes have been of limited concern. MoS₂ has been a hydrodesulphurization catalyst [de Beer, V. H. J et al., Journal of Catalysis 1976, 42, 467-470; Paul, J.-F et al., J. Phys. Chem. B 2003, 107, 4057-4064; Tye, C. T et al., Topics in
30 Catalysis 2006, 37, 129-135] which requires the edge sites to be catalytically active. The process by itself leaves the overall structure chemically unchanged although transient changes occur during the hydrodesulphurisation event. From various investigations, it is now clear that the

chemical properties of nanoscale materials can be distinctly different from the bulk and such reactivity could make inorganic analogues of graphene to be new reagents [Chen, Z et al., The Journal of Physical Chemistry C 2013, 117, 9713-9722; Mondal, B et al., Nanoscale 2016, 8, 10282-10290].

5 Water is becoming increasingly contaminated by wide variety of pollutants mainly from agricultural and industrial sources. Among all these, heavy metal contamination in water is a worldwide concern, because of their immense toxicity. Thus, to access free water, removal of heavy metals from water becomes essential. Lead toxicity and its adverse health effects are well known. The lack of understanding the risks involved in the use of lead resulted in its widespread
10 utilization, due to which plumbism came as an aftermath in many societies. There is even a hypothesis that suggest that lead poisoning might have shepherd the decline of Roman Empire, where lead pipe networks were used for water distribution [Nriagu, J.O, The New England Journal of Medicine 1983; 308:660-663]. Apart from being present as a contaminant in various water sources, lead is also a common pollutant in air along with other heavy metals like
15 cadmium and mercury. Depending on the level of exposure, lead causes a variety of diseases mainly affecting the nervous system. So, its removal from the environment is as essential as other toxic heavy metals.

Many techniques have been employed such as ion exchange, electrochemical process, membrane separation, adsorption, chemical precipitation etc., for heavy metal remediation from water.
20 Among all these techniques, adsorption is the most promising one because of its ease of operation, simplicity in design and low cost. In this context, the use of new materials is becoming very essential.

With this objective, the present invention explored the difference in chemical reactivity of bulk MoS₂ (b-MoS₂) versus its chemically exfoliated nanoscale analogue (ce-MoS₂), with
25 Pb²⁺ and Pb⁴⁺ in solution. While b-MoS₂ turned out to be completely unreactive, ce-MoS₂ rapidly transformed to PbMoO_{4-x}S_x in a reaction utilizing hydroxyl ions in solution. We show such reactions of ce-MoS₂ anchored on oxide supports can be effective means to scavenge Pb²⁺ and Pb⁴⁺ in solution, creating novel media for heavy metal remediation in waste water.

SUMMARY OF THE INVENTION

The present invention relates to a nanoscale forms of MoS₂ used for removal for toxic lead in solutions. More particularly, the present invention explored the difference in chemical reactivity of bulk MoS₂ (b-MoS₂) versus its chemically exfoliated nanoscale analogue (ce-MoS₂), with Pb²⁺ and Pb⁴⁺ in solution.

In one embodiment, the invention illustrates synthesis of chemically exfoliated MoS₂ nanosheets (ce-MoS₂NSs). The process of reacting nanoscale ce-MoS₂ nanosheets anchored on oxide support with Pb in solution at room temperature whereby the reaction is rapid and spontaneous resulting in the formation of PbMoO_{4-x}S_x. This novel reaction can be used for scavenging Pb²⁺ and Pb⁴⁺ in solution.

DETAILED DESCRIPTION OF DRAWINGS

Figure 1 (A) Representative TEM image of ce-MoS₂ NSs. The optical absorption spectrum is shown in the inset. (B) HRTEM image of a NS. Corresponding FFT pattern is shown in the inset. Lattice plane is marked (C) Raman spectrum of bulk MoS₂ and NSs. (D) Schematic of the overall reaction between ce-MoS₂ and Pb ions.

Figure 2 Characterization of the reaction product of MoS₂ and Pb²⁺ ions. (A) Standard peaks of PbMoO₄ are plotted along with the observed XRD pattern before and after the hydrothermal treatment of the reaction product. (B) SEM image showing polyhedron morphology of PbMoO_{4-x}S_x. Inset shows SEM image of one of such polyhedron. (D) Raman spectrum; TEM and HRTEM image of the same is shown in (C) and (E), respectively. A particular area of image E is marked and magnified version of that area is shown in F.

Figure 3 Characterization of final reaction product (MoS₂ + Pb⁴⁺ ions). (A) PXRD pattern of the reaction product before and after the hydrothermal treatment. SEM image at different magnifications are shown in (B, C). (D) Raman spectrum of the final reaction product. (E, F) TEM and HRTEM image of the same, respectively.

Figure 4 Conversion of microparticles to nanoparticles by mechanical grinding of the final reaction product. Standard peaks of PbMoO₄ plotted with the XRD pattern of the reaction product before and after the mechanical grinding. Inset shows the photograph of the product after grinding. (B) Raman spectrum of the same showing all the characteristic vibrations. (C, D) TEM

and HRTEM image of such particles, respectively. (E) Magnified HRTEM image of one of such particles; lattice distance is marked.

Figure 5 XPS data to support the formation of $\text{PbMoO}_{4-x}\text{S}_x$. A (i-ii), B (i-ii), C (i-ii) and D (i-ii) correspond the XPS in the Mo 3d, Pb 4f, S 2p and O 1s regions, before and after the hydrothermal treatment, respectively.

Figure 6 Equilibrium Pb^{2+} batch adsorption data, fitted using Freundlich isotherm (i, ii) Adsorption isotherm using only alumina and silica, respectively. Pb^{2+} removable capacity of $\text{Al}_2\text{O}_3@\text{ce-MoS}_2$, $\text{SiO}_2@\text{MoS}_2$ are shown in (iii, iv), respectively.

Referring to the drawings, the embodiments of the present invention are further described. The figures are not necessarily drawn to scale, and in some instances the drawings have been exaggerated or simplified for illustrative purposes only. One of ordinary skill in the art may appreciate the many possible applications and variations of the present invention based on the following examples of possible embodiments of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

The following description is presented to enable any person skilled in the art to make and use the embodiments, and is provided in the context of a particular application and its requirements. Various modifications to the disclosed embodiments will be readily apparent to those skilled in the art, and the general principles defined herein may be applied to other embodiments and applications without departing from the spirit and scope of the present disclosure. Thus, the present invention is not limited to the embodiments shown, but is to be accorded the widest scope consistent with the principles and features disclosed herein.

The present invention explored the difference in chemical reactivity of bulk MoS_2 (b- MoS_2) versus its chemically exfoliated nanoscale analogue (ce- MoS_2), with Pb^{2+} and Pb^{4+} in solution. While b- MoS_2 turned out to be completely unreactive, ce- MoS_2 rapidly transformed to $\text{PbMoO}_{4-x}\text{S}_x$ in a reaction utilizing hydroxyl ions in solution. We show such reactions of ce- MoS_2 anchored on oxide supports can be effective means to scavenge Pb^{2+} and Pb^{4+} in solution, creating novel media for heavy metal remediation in waste water.

The following sections provide the detailed description of a method of synthesizing MoS_2 NSs, the proposed driving mechanism behind the interaction of lead ions and MoS_2 NSs, in an aqueous medium, and its application in heavy metal remediation in waste water. As the

science will happen irrespective of the medium, the same will be the chemistry in a non-aqueous medium as well.

Synthesis of chemically exfoliated MoS₂ nanosheets:

5 The synthesis of chemically exfoliated MoS₂ nanosheets (ce-MoS₂NSs) was carried out using a conventional chemical exfoliation method [Coleman, J. N., et al. Science (Washington, DC, U. S.) 2011, 331, 568-571]. In an inert atmosphere of argon, 3 mL of 1.6 M n-butyllithium in hexane was added to 300 mg of bulk MoS₂ (powder) taken in a round-bottom flask. The system was left with constant stirring for nearly 48 h, maintaining the inert atmosphere. After 48
10 h, the lithium intercalated product (LixMoS₂) was collected and washed repeatedly with hexane to remove the unreacted n-butyllithium. 100 mL of distilled water was then added to this intercalated material and the system was sonicated for 1 h using a bath sonicator. Finally, this aqueous dispersion of MoS₂ NSs was centrifuged at 18000 rpm for 15 minutes to remove the unexfoliated MoS₂ as a precipitate. The supernatant containing the ce-MoS₂ NS was used for
15 reactivity studies.

Reaction of ce-MoS₂ NSs with Pb²⁺:

4 ml of as prepared MoS₂ NSs dispersion in water (4.2 mM, in term of Mo concentration) was taken in a reaction bottle, to which 1 mL (23 mM) of lead acetate solution was added. A
20 white precipitate was observed immediately, upon the addition of Pb(OAc)₂. The reaction was continued for 6 h under constant magnetic stirring and was monitored by UV/Vis spectroscopy. The reaction mixture was then centrifuged at 3000 rpm for 5 min. The precipitate was collected and washed repeatedly with H₂O to remove the excess reactants. Concentration-dependent reactions were performed by keeping the ce-MoS₂ concentration same (4 mL, 4.2 mM) and
25 varying Pb (OAc)₂ concentration (23 mM, 46 mM, 66 mM and 92 mM).

Reaction of ce-MoS₂ NSs with Pb⁴⁺:

Reaction with Pb⁴⁺ was done in a similar fashion as in the case of Pb²⁺, except for the use of a mixed solvent (H₂O: acetic acid 1:1) as Pb⁴⁺ source used (PbO₂) is not soluble in H₂O.

30 The present invention shows that the chemical exfoliation of MoS₂ NSs involved two steps. The first step involved the intercalation and in the second step, the intercalated product

was exfoliated in water. The quality of the NSs was examined using various spectroscopic and microscopic techniques. Typically, ce-MoS₂ NSs were of 200 to 1000 nm in length and had an average thickness of 2-3 layers (Figure 1a). The UV-visible absorption spectra (inset of Figure 1a) of the ce-MoS₂ NSs dispersion, shows the characteristic peaks at 435 nm, 611 nm, and 668 nm, [Wang, K., et al., ACS Nano 2013, 7, 9260-9267; Liu, H.-L Appl. Phys. Lett. 2014, 105, 201905/1-201905/4] of which the two features between 600-700 nm are known to arise from the direct transitions happening at the K point of the Brillouin zone [Mac, K. F et al., Phys. Rev. Lett. 2010, 105, 136805/1-136805/4]. The HRTEM image (Figure 1b) of the ce-MoS₂ NSs showed well resolved hexagonal lattice structure and a lattice spacing of 0.27 nm correspond to the d(100) plane [Yuen, L et al., Chem. Commun. (Cambridge, U. K.) 2016, 52, 529-532]. The Fast Fourier Transform (FFT) pattern of the HRTEM image (inset, Figure 1b) showed the expected hexagonal pattern for graphenic equivalents.

The quality of the NSs was further probed using Raman spectroscopy. On comparing the Raman spectra (Figure 1c) of bulk MoS₂ with ce-MoS₂, we find that the peak difference of A_{1g} and E_{2g} modes had decreased (~19 cm⁻¹) in the later. This difference corresponds to a thickness of approximately 2-3 layers in n-MoS₂. Also the FWHM for the A_{1g} mode has increased in n-MoS₂ (Fig. 1C) suggesting a successful exfoliation of b-MoS₂ [Wang, K., et al., ACS Nano 2013, 7, 9260-9267; Lee C. et al., ACS Nano 2010, 4, 2695-2700].

The reaction products between Pb²⁺/Pb⁴⁺ and MoS₂, in its bulk and 2D nanoscale forms, were investigated. Solutions of Pb²⁺ and Pb⁴⁺, added to the b-MoS₂ (powder) separately, remained unreactive for an extended period of time without any visible change in either the color of the dispersion or the morphology and chemical composition of the MoS₂ particles. The inactiveness of b-MoS₂ towards lead ions can be explained by thermochemical values. In stark contrast to this behaviour of b-MoS₂, ce-MoS₂ showed an immediate reaction with both the ions. Upon mixing lead ions (both Pb²⁺ and Pb⁴⁺, separately) with ce-MoS₂ dispersion, an immediate visual change was noticed. The light green color of the ce-MoS₂ dispersion turned milky white, followed by immediate precipitation of the reaction products. The white precipitate was collected and washed several times with water to remove any unreacted reactants. Both the ions (Pb²⁺ and Pb⁴⁺) gave the chemically identical reaction products, however, with different morphologies (Figure 1D). The white precipitate obtained through the reaction of Pb²⁺ with ce-MoS₂ NSs was subjected to powder X-ray diffraction (PXRD) analysis (blue trace, Figure 2A). The PXRD

pattern of the product was similar to the standard PXRD pattern of PbMoO_4 , along with a set of other peaks. These extra peaks are marked with an asterisk (*). We presumed that these peaks originated from the presence of other phases, probably metastable ones, as these could not be matched with any known phases containing the elements.

5 Pb, Mo, O, and S, as confirmed from EDS spectroscopy. To test our hypothesis, the material was hydrothermally treated at various temperatures with the expectation that these metastable phases will transform into a single, thermodynamically stable phase. An exact match with PbMoO_4 in PXRD pattern was observed after 24 h of hydrothermal treatment of the product at 190°C (black trace, Figure 2A), proving our hypothesis to be correct. The morphological
10 characterization of this hydrothermally treated product was carried out using an SEM, which confirmed that the size of the microcrystals is of the order of a few microns with polyhedron morphology (Figure 2B). The product was further characterized using Raman spectroscopy in the range from 100 to 1000 cm^{-1} (Figure 2D) [Vilaplana, R et al., J. Appl. Phys. 2012, 112, 103510/1-103510/10]. The Raman spectrum of the product was dominated by one stretching
15 mode A_g^3 at 868.8 cm^{-1} . The two peaks at 767.3 cm^{-1} and 745.7 cm^{-1} can be assigned to anti-symmetric stretching B_g^5 and E_g^5 vibrational modes, respectively. Raman peaks at 351.3 cm^{-1} and 319.1 cm^{-1} correspond to the weaker B_g^4 mode and stronger A_g^2/B_g^3 modes, respectively. The peaks at 197.1 cm^{-1} and 170.2 cm^{-1} were assigned to E_g^3 and A_g^1 modes. TEM image of the product was taken at different magnifications, for further characterization (Figure 2C, E, F).
20 HRTEM image (Figure 2E, F) of the product showed the lattice spacing of 0.33 nm corresponding to the (112) plane which further confirmed the formation of the product. EDS intensity map was performed to confirm the presence of all the expected elements. Very low intensity of S in the EDS intensity map with high intensity of Pb, Mo, and O, presumably corresponded to replacement of O positions with S in the PbMoO_4 lattice. However, this low
25 degree of S doping does not seem to affect the crystal structure as well as the interplanar distances.

Reaction with Pb^{4+} creates $\text{PbMoO}_{4-x}\text{S}_x$ directly although the particles are much smaller making a broad PXRD. The morphology of the product was completely different in the Pb^{4+} case. To confirm whether this product is indeed the same product as in the Pb^{2+} case with
30 different morphology, the final reaction product was subjected to PXRD after washing with a mixed solvent (H_2O : acetic acid). The obtained PXRD pattern was an exact match with PbMoO_4 .

The product then was also subjected to hydrothermal treatment at conditions similar to the previous case. Subsequently, PXRD measurement was done. All the peaks of the product became sharper without the disappearance of any, suggesting an increased crystallinity in the product after hydrothermal treatment. SEM images at different magnifications showed the rice grain kind of morphology of the final product (Figure 3B-C). Raman spectrum of the product revealing the same feature as in figure 2D is not identical. The blue shift in A_g^3 and B_g^5 vibrational mode compare to A_g^3 and B_g^5 mode in $\text{MoS}_2 + \text{Pb}^{2+}$ case suggesting reduced particle size and nanocrystalline nature (Figure 3D). These may be inferred from HRTEM image in Figure 3E, with a lattice spacing of 0.33 nm corresponding d(112) plane.

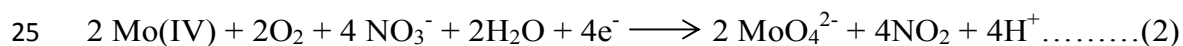
In order to further investigate the final product with rice-grain kind of morphology, mechanical grinding, of the same, was done using mortar and pestle. During the grinding, a visible colour change was observed from white to black in the inset of Figure 4A. A broad feature was seen in the PXRD of the product (Figure 4A). We attribute this to the formation of small particles. The formation of NPs within the range of around 5-50 nm was further confirmed by TEM image (Figure 4C). The HRTEM images showed the lattice spacing of 0.33 nm which confirmed that no chemical change has occurred during the grinding (Figure 4D-E). Further, it was confirmed by the Raman spectrum which showed all the characteristic peaks of PbMoO_4 .

In order to further confirm the formation of the final product, the chemical state of the elements were analyzed using XPS (Figure 5 A-D) [Du, W et al., Applied Surface Science 2015, 328, 428-435]. The XPS survey spectrum showed that the final product consisted of all the expected elements (Pb, Mo, O, and S) and no impurity peaks were detected. The specific scan in the Mo 3d region exhibited the four peaks corresponding to the presence of two types of oxidation states. The peaks at 229.3 eV and 232.4 eV correspond to the $3d_{5/2}$ and $3d_{3/2}$ Mo(VI) state, respectively. But the disappearance of one oxidation state of Mo 3d region was observed after hydrothermal treatment corresponding to the formation of a single phase in the final product. The peaks at 232.1 eV and 235.2 eV were assigned as $3d_{5/2}$ and $3d_{3/2}$ of Mo (VI) in the final product (Figure 5A). The peaks at 138.7 eV and 143.5 eV were attributed to $4f_{7/2}$ and $4f_{5/2}$ of Pb(II) in $\text{PbMoO}_{4-x}\text{S}_x$ (Figure 5B). Another doublet centered at lower binding energy (135.8 eV and 140.2 eV) were assigned to $4f_{7/2}$ and $4f_{5/2}$ of Pb(II) arising due to the presence of another binding site. Figure 5D showed the XPS at the O_{1s} region with a peak centered at 529.9 eV. Like the final product, the material before the hydrothermal treatment also showed one peak in the O_{1s}

region. The presence of dopant S was further confirmed by scanning the S 2p region (Figure 5C). The two peaks at 161.6 eV and 162.9 eV were assigned to 2p_{3/2} and 2p_{1/2} of S²⁻ in PbMoO_{4-x}S_x. The chemical reaction involves the conversion of Mo(IV) to Mo(VI) as revealed by XPS. This required the involvement of oxygen. In order to test the origin of oxygen, the reaction was performed at various pH. The reaction occurred only in basic medium. The hydroxyl ions present in the reaction medium are driving the reaction and the decrease in pH during the reaction has confirmed the origin of oxygen in the product. We propose a chemical reaction in which Mo(IV) was transformed to Mo(VI) driven by OH⁻ ions while OH⁻ formed H₂O and O₂.



Although the reaction is thermochemically nonspontaneous, it can be driven forward due to the involvement of metal ions. We conjecture that acetate ions (coming from lead acetate) are getting reduced to ethanol during the course of the reaction to balance the charge. To prove this, the same reaction was done taking lead nitrate as a precursor and the formation of NO₂ was examined by *in situ* mass spectrometric detection. Both the reactant solutions were purged with Ar to remove the dissolved O₂, separately. Then the lead nitrate solution was injected into the MoS₂ dispersion taken into an enclosed vessel. The mixture of gases inside the vessel were analysed after 2 h reaction using a residual gas analyzer. An increase in ion current was observed for both NO₂ and N₂ due to the increase in partial pressures of the corresponding gases. When the control (MoS₂ dispersion without lead acetate solution) was analysed, reduced ion current for NO₂ was observed while ion current for N₂ (background gas) remained the same. The control and sample were analysed under identical reaction conditions to account for some unavoidable leakage. So, the overall reaction for the second and third step can be written as follows:



The experiment suggests the formation of an acid which was again supported by the decrease in the pH during the course of the reaction. With tap water, it was observed that as long as lead ions are present, these reactions do occur upon introducing n-MoS₂.

This unusual reactivity can be used for the capture and removal of lead ions from water. In a typical batch experiment, 50 mg of ce-MoS₂ adsorbed (0.5 mL, 4.2 mM, in term of Mo

concentration) on alumina or silica was taken in a 200 mL of conical flask containing 50 mL of Pb^{2+} solution. The removal% and uptake were calculated using the equations mentioned below:

$$\text{Removal\%} = \frac{C_o - C_e}{C_e} \times 100$$

$$\text{Uptake (q}_e) = \frac{(C_o - C_e)V}{m}$$

5 Where C_o and C_e are the initial and equilibrium concentrations of the metal ions, respectively, V is the volume of the solution (L) and m is the mass of the adsorbent (g). The removal capacities of $Al_2O_3@ce-MoS_2$ (282 mg/g) and $SiO_2@MoS_2$ (199 mg/g) nanocomposites for Pb^{2+} were evaluated using the Freundlich adsorption isotherm. A plot of $\log q_e$ (q_e = heavy metal uptake) vs $\log c_e$ (c_e = equilibrium concentration of heavy metal ion) showed a straight line with intercepts
10 0.97, 0.68, 2.45 and 2.3 and slopes 0.47, 0.63, 1.05 and 0.80 for Al_2O_3 , SiO_2 , $ce-MoS_2@Al_2O_3$ and $ce-MoS_2@SiO_2$, respectively. Batch experiments were performed with different initial concentrations ranging from 1 ppm to 200 ppm. The data were then fitted using the linear form of Freundlich adsorption isotherm, $\log q_e = \log k_f + \frac{1}{n} \log C_e$, where k_f is the amount of heavy metal ions adsorbed per g of adsorbent (mg/g). Although Freundlich isotherm is not perfectly
15 valid due to the chemical reaction between MoS_2 and Pb^{2+} ions, such models were used in similar case where MoO_3 reacted with Pb^{2+} but still Langmuir and Freundlich adsorption isotherm were applied to calculate the adsorption capacity [Wu, Y et al., Journal of Colloid and Interface Science 2017, 491, 80-88].

20 It may be appreciated by those skilled in the art that the drawings, examples and detailed description herein are to be regarded in an illustrative rather than a restrictive manner.

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We Claim:

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1. A process of scavenging Pb^{2+} and Pb^{4+} in a solution, the said process comprises
 - a. adding 1ml of lead (Pb) source to 4ml of chemically exfoliated-MoS₂ nanosheet anchored on oxide support in a reaction bottle and constantly magnetic stirring the reaction for 6 hours;
 - b. centrifuging the reaction mixture at 3000 rpm for 5 min; and
 - c. collecting the precipitate and washing repeatedly with H₂O to remove the excess reactants

wherein, the said scavenging of Pb^{2+} and Pb^{4+} occurs through a chemical reaction between the chemically exfoliated-MoS₂ nanosheet and lead in the solution resulting in an instant visual change, where light green color of the chemically exfoliated-MoS₂ dispersion immediately turns milky white upon reacting with the lead in solution, followed by precipitation of the reaction products in the form of PbMoO_{4-x}S_x complex.

2. The process as claimed in claim 1, wherein the MoS₂ nanosheets is in the size up to 1 μ m in length with 2-3 layers thickness.
3. The process as claimed in claim 1, wherein Pb is in Pb^{2+} and Pb^{4+} state.
4. The process as claimed in claim 1, wherein the lead (Pb) source used in the process for Pb^{2+} is Pb(OAC)₂ and for Pb^{4+} is PbO₂.
5. The process as claimed in claim 1, wherein the MoS₂ nanosheets are supported on oxide including SiO₂, Al₂O₃.
6. The process as claimed in claim 1, wherein the oxide supported chemically exfoliated-MoS₂ nanosheets are used as an adsorbent to capture and remove the Pb species from water.
7. The process as claimed in claim 1 wherein the solution is aqueous or non aqueous.

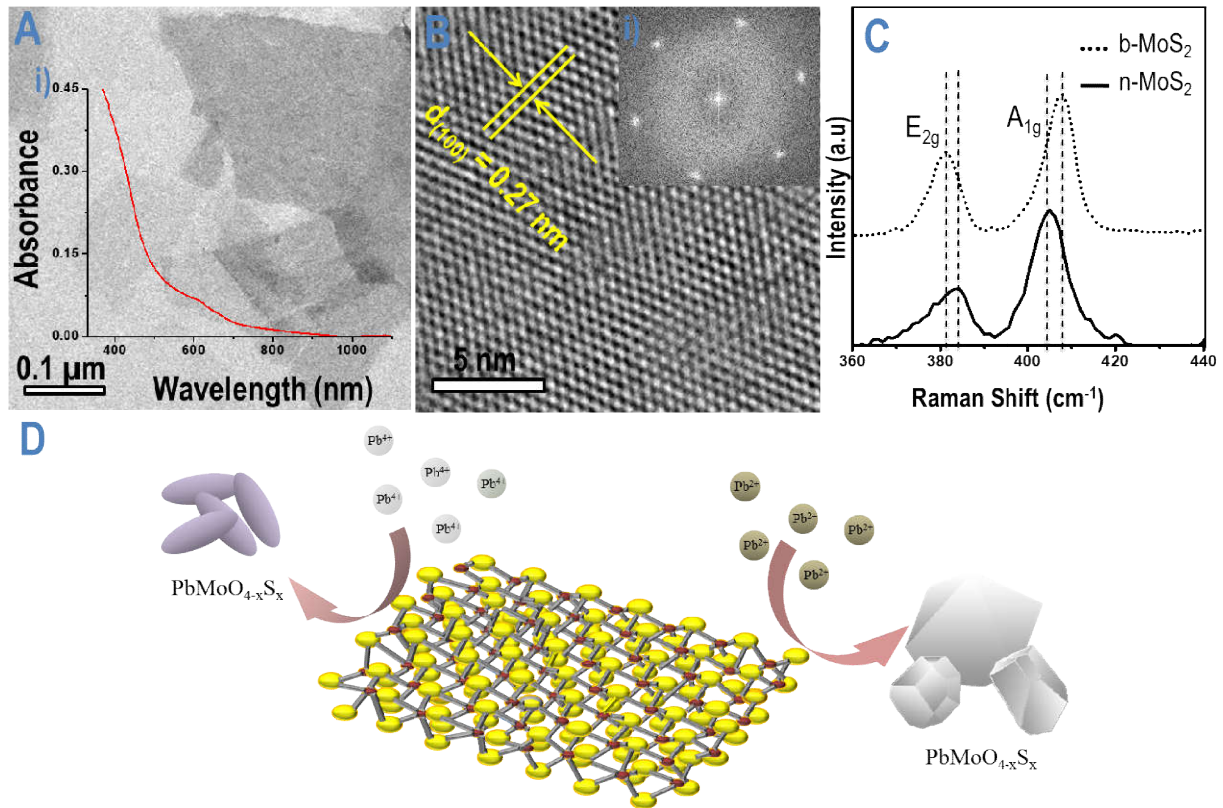
Dated at Chennai this January 29, 2020

Signature: 

D. Moses Jeyakaran
Advocate & Patent Agent
IN/PA — 369

REMOVAL OF LEAD FROM WASTE WATER USING NANOSCALE MoS₂

5



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FIGURE 1

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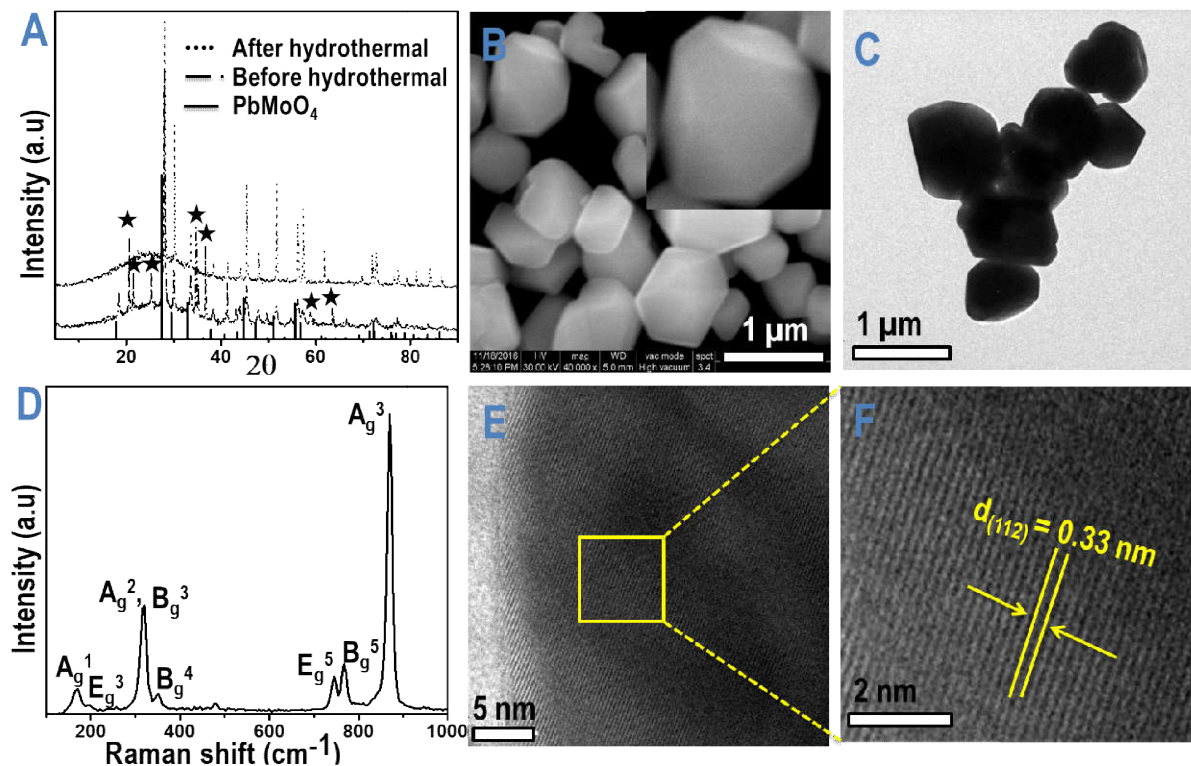
Signature: 

D. Moses Jeyakaran
Advocate & Patent Agent
IN/PA — 369

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REMOVAL OF LEAD FROM WASTE WATER USING NANOSCALE MoS₂

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FIGURE 2

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Signature: *D. Moses Jeyakaran*

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D. Moses Jeyakaran
 Advocate & Patent Agent
 IN/PA — 369

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ABSTRACT

REMOVAL OF LEAD FROM WASTE WATER USING NANOSCALE MoS₂

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The process of reacting nanoscale ce-MoS₂nanosheets anchored on oxide support with lead in solution at room temperature whereby the reaction is rapid and spontaneous resulting in the formation of PbMoO_{4-x}S_x in the process of scavenging Pb²⁺ and Pb⁴⁺ present in the solution.

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