CHEMICALLY DERIVED TRANSITION METAL DICHALCOGENIDES MoS_2 BASED THIN FILMS.

DISSTERTATION REPORT SUBMITTED

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Shubham

TABLE OF CONTENTS:

INTRODUCTION:

Thin films [1-2] are demands of modern science and have revolutionized modern technology. Thin films are defined as, layer of materials whose size is lesser than one micrometre to several nanometres in thickness. The synthesis of materials thin films have been done by a number of methods such as Spin Coating [1-2, 5], Sol-Gel [1-2, 5], Dip-Coating [1, 2, 5], Chemical vapour deposition (CVD) [1-2, 5, 23], Microwave Annealing [10, 18] and many more. These can be characterise by various methods of characterisation like X-Rays Diffraction (XRD) [1-2, 5], Fourier Transform Infrared Spectroscopy (FTIR) [1-5], Scanning Electron Microscopy (SEM) [1-2, 5], Transmission Electron Microscopy (TEM) [1-2, 5], Ultra-violet Visible Spectroscopy [1-5], Raman Spectroscopy [1-5], and many others. Working on such a small scale is so much helpful as it provides numerous advantages and profits like fabrication of electronic semiconductor devices, optoelectronic devices, and memory storage devices etc. On the same way, a huge advantage in aspects of efficiency is easily approachable while working with the devices which are formed on a small level or scale (micro to nano level) from various compounds of different materials. Such films get the credit for playing an essential part in intensifying and developing study of materials to new and unique level, as countless properties of the materials can be extracted. Furthermore the material science is growing day by day therefore many numbers of materials compounds have discovered whose nano films have been synthesised so far, likewise thin films of transition metal di-chalcogenides are one of those and their novel characteristics lead wide development in today's science and technology. Talking about the current area of fascination in research, is semiconducting thin films of tungsten disulphide (WS_2), tungsten diselenide (WSe_2), molybdenum diselenide ($MoSe_2$), molybdenum disulphide (MoS_2) and their various

composites. They can be announced as the backbone of electronic industries in this current modern era.

WHAT IS MoS_2 [5, 24]:

The molybdenum disulphide is an inorganic compound which is strong competitor to graphene, having chemical/molecular formula MoS_2. In general, it is defined as MX_2, where, M stands for transition metals and X stands for chalcogenides. Its colour composition in appearance is silvery black or kind of lead-grey. Moreover the valency of molybdenum is +4 and sulphide is -2 in MoS_2 and it is prepared in single layered as well as multi-layered with a sandwich kind of sequence i.e. (S=Mo=S). The nature of bonding in between Mos_2 layered compounds are covalent and Van-der Waal. Monolayer of MoS_2 is having direct band gap of range 1.8-1.9 eV, depending on purity of prepared sample on the other hand, bulky or multilayer of such compound is indirect band gap of 1.2eV which corresponds to various electronic properties of this material such as formation of semiconducting device like transistors, photodetectors, and memory storage or resistive memory etc. The semiconducting properties corresponds to its trigonal symmetry whereas, metallic properties corresponds to octahedral geometry in crystalline form. It is a two dimensional material and hardly soluble in dilute acid as well as in polar solvents like water, ethanol and so on, but it is soluble in hot acids like H_2SO_4, HNO_3. Therefore, these properties of MoS_2, introduce an attractive area of research.

LITERATURE SURVEY:

1. BRANIMIR RADISAVLJEVIC AND ANDRASKIS [7]:

This paper reveals the application part of monolayer $MoS_{2,}$ flakes of which are exfoliated from the bulk molybdenite crystals. Quantum mechanical confinement of single layer MoS_2 offers so many promising properties from nano-electronics to sensing and photonics too. But, presence of direct band gap of monolayer MoS_2 can work as a channel for field effect transistors (FET) with high on/off ratio of current. Further the high k-dielectrics reflect one more dominating properties of it, is high mobility of charge carriers through these i.e. device fabricated from MoS_2 flakes and these works as gate in FET formed. Also the effect of temperature on the mobility of charge carriers has been studied which reveals the dependence of mobility on applied temperature and concluded that both have linear relationship with each other. On the same hand, doping effects are also studied on single layer MoS_2 which allow the observation of metal-insulator transition through it and main cause of it is strong electron-2 interaction.

2. NAR DEEP KUMAR et al. [8]:

This article shows results regarding the effect of lacking of symmetry element (inversion symmetry in monolayer) of TMDCs especially thin films of MoS_2. Numerous researches has been done on MoS_2, most appropriately available properties like mechanical, electronic, linear optical and many more but the non-linear optical responses are not promptly known. This paper mainly depicts those effects which are main prospect in matter- light interaction and have widely fruitful applications in optoelectronics, laser pulses and photonics. The bulk crystal of MoS_2 belongs to space group D_{6h} having 2H stacking order. As broken of inversion symmetry gives huge effect that decreases the

working of inter-band transitions which are valley-selective. Consequently allows optical second harmonic generation, due to which 820-nm pulse is generated in exfoliated monolayer of MoS_2 mechanically. During this kind of deformation the magnetic non-linear susceptibility is of order 10−7 m/V. A sample is prepared by chemical vapour deposition technique at a temperature range from 750 ^{0} C to 850 ^{0}C. The prepared MoS_2 which is having triangular shape is deposited on a substrate of Si/SiO_2. After this, measurements are done by second-harmonic microscopy measurement that beautify many number of merits in detection of crystal orientation, stacked layer domain size of single crystalline thin films of MoS_2.

3. D. J. SATHE et al. [9]:

In this research article, the main work is the thin film synthetisation of molybdenum disulphide. The method used for this purpose is a chemical bath, in which MoS_2 is prepared by reduction of Mo^{6+} ions to Mo^{4+} ions. This is done by the reaction of Mo^{6+} with citric ion and then with hydrazine which results in its reduction but temperature should be 298 K during the deposition. Similarly, S^{2-} ions are formed by reduction of sodium thiosulphate. In this process the most important process parameter is the bath temperature which should be taken into consideration during the deposition. To prepare the nano film of MoS_2 a condensation process has been done. In this process, MoS_2 particles are converted from solid phase to condense phase, then the condensed particles are deposited on a glass substrate in a chemical bath machine with rpm of 60-70 at room temperature. The thickness of the formed film is calculated by gravimetric method and other characterization like for symmetry XRD is done which gives intensity peaks at (300), (410), (600), (602), and (801) justifying their single phase property. Furthermore EDAX gives stoichiometry data of thin film whereas UV analysis gives the value of absorptivity 1.84×10^4 cm^{-1} from which band gap is calculated i.e. by plot of absorptivity

vs photon energy and it comes to be 1.7 eV which is fairly match with theoretical data of MoS_2. Such a high value of absorptivity of this material proves its high applicability in opto-electronic devices.

4. DUCK HYUN YAUN et al. [10]:

A new type of device fabrication is done by using a technique named as microwave annealing for sample synthesis. Mainly the nanocrystals of MoS_2 are prepared on graphene sheets which work as electrode in batteries, solar cell, capacitors and many others. The technique is so easy and convenient for the synthesis purpose of sample material and it is called as hybrid microwave annealing. MoS_2/GR (* here GR stands for reduced graphene *) sheets are directly prepared in few minutes of heating of solution containing graphene oxide, $MoCl_5$ and ethanol in microwave at 1000 watt of power but main thing which should be taken under consideration, that there is no direct heating of solution in microwaves. But it is to be done under a graphite block which works as microwave absorber through which heating is send to sample solution by usual heating transfer mechanism which results in reduction in graphene oxide to graphene directly and also the formation of MoS_2 nanoparticles by further heat transferred by reduced graphene. By this way MoS_2/GR nanocomposite is formed. This synthesis method is more or less resembled with sintering ceramics. The technique used for the synthesis of nanoparticles is very fast and take time less than one minute for sample preparation. XRD characterisation gives the conformity of layered structures by giving the peak (002) at 14^o and the presence of reduced graphene is assure due to absence of peak at 11^o. Furthermore TEM and SEM images provides morphology and topographic data of sample and Raman spectra gives data regarding degree of order and disorder in sample due to GO-Mo-thiourea complex at different temperatures whereas complex to MoS_2 upon hybrid microwave annealing is given by two major peaks one in plane mode (382 cm^{-1})

and another is out of plane mode (408.5 cm^{-1}) which confirms the formation of MoS_2/GR. Hence the synthesised material is not only helpful in electronics, optoelectronic or semiconductors industries but also helpful as electro-catalyst as per chemistry point of view because from above method the hydrogen evaluation reaction is also occurring i.e. reduction of graphene oxide to graphene which is quite a good alternative for scarce platinum. So this method is very efficient for fabrication graphene based hybrid materials in area of research and applications.

5. CHAOLIANG TAN et al. [11]:

This paper depicts, the results regarding unique properties of transition metal di-chalcogenides having varying properties from their single layers to bulk layers. For instance MoS_2.TeS_2, TiS2 i.e. combination of different transition metals with sulphur (which is a chalcogen) results in unique kind of nano sheets composite of two dimensions and have promising applications in sensors energy storing devices as well as in many other electronic device fabrication . Such a sample is prepared in aqueous solution form using electrochemical Li –interaction method and is characterised by using SEM, TEM and AFM. These formed thin nano sheets are investigated and observed that which are quite helpful in enhancing the fluorescence of aggregate induced emission AIE molecules which come under the categories of fluorophores. The effect of thin single layer MoS_2 nano-sheet on fluorescence of AIE was investigated using fluorescence microscopy in reference with N, N-diphenyl-1, 3, 5-triazin-2-amine (DDTA). Coating of single thin layer of MoS_2 on DDTA results in enhanced fluorescence or PL intensity as compare to without its nano-sheets. The whole structural and surface studies of DDTA with or without MoS_2 are done by using transmission electron microscopy (TEM). So the thin

layer put great impact on such organic samples and can be used as Light responsive Sensors, Biological probes and many more.

6. MING-YANG LI et al. [12] :

This paper demonstrates formation of lateral p-n junction by epitaxial growth of monolayer of two dimensional transition metal di-chalcogenides for example WSe_2-MoS_2. As they work on epitaxial growth of TMDs in which the growth of WSe_2 on a substrate is done by Van der Waals epitaxy and side by side followed growth of MoS_2 by edge epitaxy. This process done in two steps that results the interface to be sharped atomically. Firstly a single layer of WSe_2 of triangular shaped is formed at temp (925 ^{0}C) and then in a separate furnace the growth of MoS_2 done at temp (755 ^{0}C) and finally sample was synthesized using chemical vapour deposition and grew on the sapphire substrate using Van der Waals epitaxy. The shape and size of inner plane is given by Photoluminescence and by Raman Spectroscopies. To distinguish different atoms as per their intensity is given by annular dark field image of the junction formed. The sample formed has immense application in electronics as it is a hetero-structure of a monolayer WSe_2-MoS_2 as various characterizations like STEM, TEM, and SEM and so on reveals results in support of high on-off current ratios and peculiar optic-electrical characteristics.

7. XUFAN LI et al. [13]:

The preparation of two dimensional layered semiconductors with combination of metal and chalcogenides like MX [where M is transition metals like Tungsten (W) or Molybdenum (Mo) and X is chalcogenide like Sulphur (S) or Selenium(Se) or Tellurium (Te)] and metal di-chalcogenides [MX_2 where M as transition metal and X_2 di-chalcogenides]. A hetero-structure is formed using different 2D materials (GaSe/$MoSe_2$)

by Van der Waals epitaxy the formed structure will have matched lattice as a result class physical properties successfully fabricated. But there is challenge to grow this hetero-structure semiconductor layer to number of hetero-structures with large lattice misfits and this resolved by two step CVD. The MX and MX_2 monolayers are vertically stacked due to that these layers show VDE and having well aligned orientation between two layers results in a kind of super-lattice. But lateral hetero-structure do not exhibit further any lateral epitaxial alignment between the layers of crystalline domains of GaSe and $MoSe_2$. On the other hand, there is same lattice orientation is observed in lateral hetero-structures layers as initially. Hence the further growth of monolayers of $MoSe_2$ is done by GaSe using CVD this result in forming a strip of vertically stacked VDE (Van der Waals Epitaxy) hetero-structures at crystal interface. This type of vertically stacked hetero-structures exhibit the semiconductor properties and a kind of p-n junctions with transport of photo generated charge carriers in between these layers and have great tuneable photovoltaic response. Their all such behaviour, come due to their properties and uniqueness in synthesis by using CVD and VDW further their all properties are studied by using various experimental techniques such AFM, TEM, SEM, XRD and calculation of crystalline properties is done by using DFT mainly. These hetero-structures have numerous applications like gate-tuneable field effect transistors, photo detectors and solar cells.

8. XIDONG DUAN et al. [14]:

This paper presents the results regarding two dimensional heterojunctions formed by Transition metal di-chalcogenides (TMDCs) for instance WS_2, $MoSe_2$, MoS_2, and WSe_2. Production of hetero-structures layers of MoS_2-$MoSe_2$ and WS_2-$WoSe_2$ formed by a chemical modulation technique in situ having vapour phase reactants during their growth.

As using lateral epitaxial growth techniques for characterisation of both $MoSe_2$ –MoS_2 and $WoSe_2$-WS_2 reveals triangular geometry. Further the structural and optical modulation has been depicted from data of characterisation using Raman and Photoluminescence studies. Moreover AFM results for WS_2-WSe_2 and MoS_2-MoS_2 gave description regarding their thicknesses which were of order 10-50 nm and 0.8-1.0 nm respectively for these layered hetero-structures. Even though chemical modulation of sulphur and selenium were opposite but TEM and EDS reports revels the single crystalline structure. Some semiconducting properties are (lateral p-n diodes) also depicted by differently layered composite of WS_2-WSe_2 and characterise through electrical transport studies. Such composites results in distinguish properties like highly responsive for current rectification and photocurrent generation which can be used to create semiconductor devices such as CMOS.

9. SHRADDHA GANORKAR et al. [15]:

In this research article they mainly depicts their work that how can the growth of monolayer, bilayer, trilayer, tetralayer MoS_2 thin films using two different precursor solution of MoO_3 and $MoCl_5$ respectively in single step reaction using distinguish experimental parameters with help of chemical vapour deposition technique. The main reason is the phase transition occurs from liquid to solid phase in this single step reaction and got different results due to different precursor solution. The reaction of MoO_3 and sulphur gives monolayer MoS_2 with a kind of triangular shaped grains which is identified using optical images and raman spectra (it gives raman shift at $\Delta k = 21.5\ cm^{-1}$) whereas photoluminescence spectra for monolayer gives absorption peak at 1.83 eV (675.73 nm) and 1.99 eV (621.78nm). Furthermore the result came after reaction of $MoCl_5$ with sulphur in optical images demonstrate monolayer and bilayer of MOS_2 without triangular

shape and characterize using raman spectroscopy (shift at $\Delta k = 21.5\ cm^{-1}$ for monolayer and for bilayer shift at $\Delta k = 22.6\ cm^{-1}$). The photo luminescence for bilayer MoS_2 give absorption peak at 1.88 eV (657.44 nm) and 2.04 eV (605.10 nm). Likewise, for monolayers the results are same as MoS_2 prepared with MoO_3. All these results clears that the $MoCl_5$ is an appropriate source for nano film preparation of MoS_2 because the film formed using this as precursor solution in more uniform than MoO_3. Thus this method is very helpful in device fabrication mainly for layered MoS_2 as it offers wonderful property of tuneable band gap.

10. <u>WENZHUO WU et al.</u> [16]:

This research letter demonstrates the experimental facts about MoS_2 flakes in different forms i.e. in single layers and bulk layers. The main content of their study is piezoelectric characteristics of this 2 dimensional material, as piezoelectric effect main characteristics of thin films, nanoparticles and bulk crystals. The different layers of MoS_2 depicts different properties if we talk about this effect on monolayer of MoS_2 which has strong piezoelectric properties whereas in bulky layers this effect do not appears, as reason behind this opposite orientations to their adjacent one's in the bulky layers. Even the experimental results show that MoS_2 flakes has piezoelectric properties due to cyclic stretching and releasing of their odd number of atomic layers. Therefore a kind of piezo voltage and current is produced due to such oscillations in odd number of atomic layers it means there conversation of mechanical energy to electric energy no result will come due to even number of layers. A small mechanical exfoliation results in strain in the single atomic layers of MoS_2 this strain increases if we decrease the thickness of these layers. The main reason of this conservation of energy is the strain induced lattice distortion and ion charge polarization in single atomic layers. There is no such distortion bulky layers,

thus these nanolayers shows an attractive application in sensors, transducers, and many other electronic devices.

11. CHAO FAN, ZHONGMING WEI, SHENGXUE YANG AND JINBO LI. [17]:

The formation of nanostructures using Molybdenum di-selenide ($MoSe_2$) can be possible it is belongs to TMDCs family. It is a type of semiconductor material having a band gap around 1.7-1.9 eV. Using this compound material-flower like nanostructures can be made with help of technique name as facile hydrothermal. For sample preparations purpose initially Na_2MoO_4 and selenium (Se) taken as reagents (precursor) in the powder form in suitable amount put in a autoclave (50 ml) of stainless steel .Further an amount of hydrazine hydrate was added to it and the autoclave filled with distilled water (till a level of 38 ml of container) and pH =12 should be maintain or adjusted by adding solid NaOH. The container was sealed properly at temp180 ^{0}C for a period of 48 hour and wait till cooling at room temperature, and the completion of reaction results in formation of black precipitate form. This crude product washed with water and ethanol and final product would undergo under dry process in a container of vacuum at temp 60 ^{0}C. The resultant product was then under go for characterization process like XRD (for crystalline property) and using SEM and HRTEM images results depict flower-like structures. From EDS measurements average atomic ratio is coming 2.6 between molybdenum (Mo) and selenium (Se). The devices formed using this material (nanostructures), are photo responsive to red illumination. The characterisation data conclude high electrical conductivity and new light (photo) responsive properties of these nanostructures.

OBJECTIVES:

1. To prepare the MoS_2 by microwave irradiation process.

2. To characterize the prepared MoS_2 samples.

3. To prepare MoS_2- PVA nanocomposite thin films and their characterization.

EXPERIMENTAL WORK:

After reviewing so many research papers and online stuff for synthesis of thin films of MoS_2 and powder form of it. I come to conclusive method for my experimental work which is one of the convenient techniques known as microwave irradiation. It is use for synthesis of MoS_2 in powder form and after that it's chemically derived nano film can be prepared by using spin coating method. The method which I am using for the sample synthesis is quite easy in the sense of handling and processing on it and takes hardly a time of 3 minutes for whole of procedure to be done after making the solutions with aptly stirring. A question arises what is that microwave irradiation, basically in this method a heating of prepared solution is done in a microwave oven where the atom or molecules get irradiated with microwaves and directly the solution (liquid form) gives the kind of powder form i.e. a phase transition from liquid to solid state directly occur without affecting the melting point of the prepared solution or liquid. This process is somehow resembles with the sintering process. How it is better than other rottenly used experimental techniques for the sample synthesis of MoS_2 like hydrothermal, chemical bath, chemical vapour deposition, silar, liquid exfoliation and so on. I am going to explain of few these techniques from that stuff an easy comparison will come in front of us that the how the method used by me for my sample synthesis is better than those other ones.

i.) Hydrothermal Method [17, 25]: As the name suggested the type of method i.e. hydro stands for water and thermal stands for heat or temperature. It is used mainly for those solvent or for the materials, which are soluble in hot water at high pressure. A special types of instrument known as autoclave is used for this process to get the proper crystal growth. The end where temperature is high i.e. hotter part where whole of dissolving process is done, and on the cooler end, crystallisation growth of the desired material is occurs. This is used for those materials which have high vapour pressure at their melting point so that to set their crystallisation phase even though the sample is not stable at this temperature that means at its melting point. So it is clearly seen that, this method in not well for the samples which I have prepared. Main reason of this is the cost of autoclave as it is too expensive besides less solubility of solution in hot water which I am using for experimental work. Hence it is giving clear picture that, this technique is not suitable for my work.

ii.) **Chemical bath [9, 22]:** It is a chemical method for which a direct synthesis of sample thin films and simplest too. This method requires a solution sample material in vessel and another requirement is the substrate on which we want to process of deposition. Adding the complexity the main complications are stirring process, maintenance of proper and constant limit of temperature, thermostat bath, ph. value and concentration of solute in the solvent all these matters a lot. This method mainly carried for alkaline solution but it does not mean that it is not applicable for acidic solution. Rise in the temperature of bath results in elevation in the ionic motilities and rate of diffusion therefore the conductivity can be calculated for the given sample. This temperature of the bath effects the crystalline growth as more the temperature more will be crystalline growth. Thus we can see although it is a simplest method for directly preparation for thin of required materials but

their too much complication and terms which we have to consider therefore this methods also lags in front of the method which I am using.

iii.) **Silar Method [21]:** It is known as successive ionic layer absorption and reaction and in this method we firstly prepare the precursor cationic solution of sample materials in a vessel. After that a glass substrate with proper cooling is put into a vessel with a constant heating so that ion of cationic solution is being absorbed by the glass substrate. Then after it is properly rinse with doubly distilled water in excess amount so extra content get dissolved water and get away from substrate then this substrate is dip into a beaker of anionic solution which prepared in appropriate pH value, further the anionic ion are being absorbed on the glass substrate and it is again rinsed with doubly distilled water. Hence a homogenous thin film of sample material is prepared after repeating cycle of this process. So the whole procedure takes lots of time with genuine care while rinsing and for making the different solutions.

iv.) Chemical Vapour Deposition [1, 5, 23]: This is one of the famous chemical deposition techniques for the deposition of thin films with desirable properties. In this technique, a substrate inside the CVD tube is exposed to a mixture of volatile gases which further gets condensed and nucleates on the substrate and makes the thin films. Mainly CVD is of three type's which are as follows: (1) high pressure CVD (2) low pressure (3) plasma enhanced CVD. The working substances used in CVD process, generally are gaseous substances. During thin films growth, this process mainly works on various pressure ranges. This process is use a lot in today's era in the various fields like in electronics, fabrics of organic devices and biological materials.

Therefore, it is clear from above explained procedure of the some of the techniques for MoS_2 preparation although these methods quite good at their level. But they offer too

many precautions simultaneously we have to take care physical as well as their chemical parameters with huge time duration. On the other hand, the method which is I am using is quite simple and steady with little precautions. One of the beautiful advantages of technique which is used by me is that it is very simple process and requires less process time and it is very low cost process.

Synthesis process of MoS_2 [10, 19, 20, 24]: For making powder form of MoS_2, before starting the synthesis procedure the all the equipment should be cleaned using ethanol and double distilled water so there will be no contamination as we working on nanoparticles formation so contamination can affect our results of 0.5gram of Molybdenum (V) chloride (Sigma- Aldrich 95%) ($MoCl_5$) was dissolved in 1.50 ml of ethanol with constant stirring for few minutes in a 50 ml beaker. The Molybdenum (V) chloride reacts very vigorously with ethanol and instantly leaving chlorine from it in the form HCl so that a dark green solution is formed shown in **Figure 1(a)**. Then after a stoichiometric amount of thiourea about 280mg is added to earlier formed solution which is used as sulphur source after that a stirring of this solution is done for that Teflon magnetic bead is put into beaker and cover the beaker with a watch glass to avoid the contamination present in atmosphere and the stirring is for about 1 hour on a magnetic stirrer so that a homogenous solution is formed. Now put the Teflon bead out of the beaker and after this prepared solution will go for microwave irradiation in a household microwave oven, **Figure 1(b)** at about 600-1400 watt of power with three sets of heating in intervals of 1 minute each at temperature range 700 ^{0}C-750 ^{0}C and suddenly a cluster yield in the form of powder will form shown in **Figure 1(d)** (* I did synthesis of samples at three different powers*) and just take the beaker out of the microwave oven and kind of powder form observed **Figure 1(f)**. Then do the cleaning of prepared powder with excess of ethanol and doubly ionized water so that insoluble product will top of the filter paper and we get our desire result.

Fig. 1(a): As prepared precursor solution

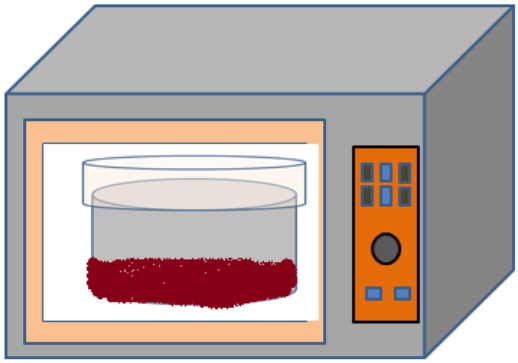

Fig. 1(b): Sample under microwaves irradiation

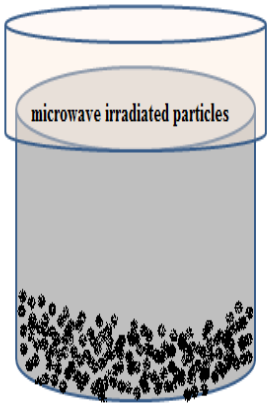

Fig. 1(c): Irradiated particles

Fig. 1(d): Yield in cluster form like a plateau

Fig. 1(e): Final yield after microwave irradiation process

Fig. 1(f): Powder form of the as prepared MoS_2

Prepared samples and their observations [10, 19]:

Sample (1): I prepared my first sample as per above described methodology but I forgot to take the bead out of the prepared solution before the microwave irradiation that result into burning of my sample and i came to know new things in front of me. When i took my sample out of microwave after heating i saw that the sample was burnt and it turned into black colour simultaneously i observed that the teflon bead was just unaffected but the magnetic inside is burnt. So it gave me a kind of lesson regarding the precaution which i have to take before the microwave irradiation. The burnt sample is shown in **Figure 2(a)** and **(b)**.

Fig. 2(a-b): Burnt material during microwave irradiation

Other samples: After learning from the first experience of sample synthetisation then I prepared three other samples at different parameters in terms of power of microwave oven. As per the methodology explained earlier and I observed the crystallisation of the samples done in few minutes as I put my precursor solution in microwave oven shown in **Figure 1(d)**. Sample (2) is prepared at 600watt (a) with three heating intervals in a microwave oven of 50 seconds each and another sample (3) prepared at same power but only difference in time for which heating has been done. Here heating in interval of 60 seconds each (b), sample (4) is prepared at 1000 watt (c) and sample (d) is prepared at

1400watt of power (c) with heating in three sets of 1 minute each then all these are collected their respective images shown below in **Figure 3(a-d)**.

Fig. (a)

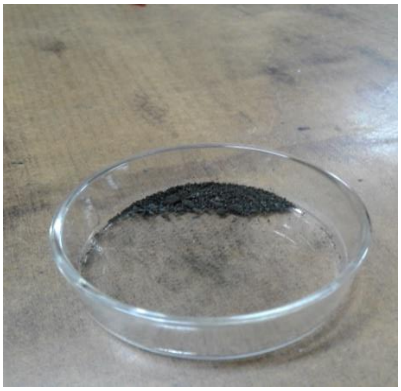

Fig. (b)

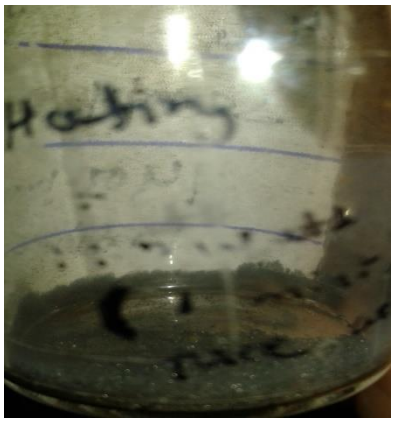

Fig. (c)

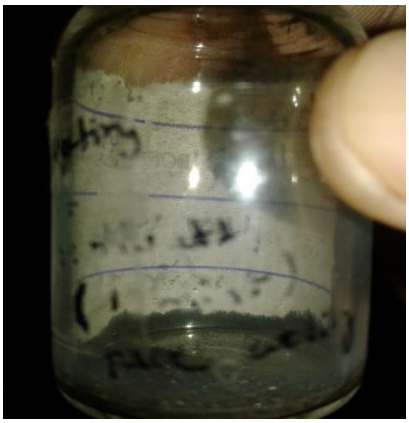

Fig. (d)

Figure 3(a-d): Shows the MoS2 sample prepared by microwave irradiation at different Watts.

Precautions and conditions: We have to take care of few things while performing this experimental work which are as follows:

1. Make sure the beakers, spatula, watch glass, petri dish should be cleaned properly before the use; otherwise it will affect the results.
2. The measurement of $MoCl_5$ should be done on a watch glass because it is a quite reactive chemical. It shows reactivity even with the paper sheet or on filter paper. On the same way, it shows too much reactivity in atmosphere so it should be free from moisture otherwise the whole chemical will demolish.
3. Make sure while making the measurements of $MoCl_5$ keeps it way from your nose and eyes, as it can cause eye damage and serious drug type of addiction. Therefore it is good if we use the gloves and mask while doing the solution preparation with these chemicals.
4. The measurements other chemical like ethanol and thiourea should be done in precise amount only otherwise it directly affects the results.
5. Prepared mixture of ($MoCl_5$ + ethanol + thiourea) should be stirred on a magnetic stirrer for 1 hour in 400-500 rotation per minute so as the final mixture become homogeneous.
6. Before the irradiation process the microwave should be calibrated properly i.e. need to warm up the machine for few minutes. After the completion of irradiation process carefully take beaker out of the microwave oven. Then a cluster of yield kind of plateau will form which is then take out of the beaker with help of spatula after making proper powder form and put into a borosil glass bottle.

Results and Discussion:

FITR characterization: After synthesis of different samples and FTIR of all these samples has been taken. In **Figure 4(a)** the ir spectra of $Mocl_5$+Ethanol is shown where the peak at 867.03 cm^{-1} is for chloride is observed and peak at 3400-3700 cm^{-1} a sharp absorption layer for -OH group is observed. This results indicates that the initial reaction is going in an appropriate way and **Figure 4(b)** for pure thiourea, N-H bending in the finger print region at 1550 cm^{-1} as well as C-N stretching is observed at 1050 cm^{-1}. Moreover N-H stretching in functional group region for symmetrical and anti-symmetrical peaks at 3300 cm^{-1} and 3400 cm^{-1} [3, 5] insuring primary amine coupled doublet. Accordingly C=S stretch peak observed at 1050 cm^{-1} [3, 5]. I have taken the ir data in **Figure 4(a) and 4(b)** because from that we will be able to analyse the shift in peaks after formation of product. Therefore in **Figure 4(c**), the spectra peaks in FTIR for as prepared sample is being analysed so clearly a change can be seen in C=S stretching from 1400-1500 cm^{-1} as compare to pure thiourea due to formation of coordination compound of sulphur with metal ions. Therefore this molybdenum and sulphur asymmetric stretching results in reduced double bond character of C=S bond side by side increase in C-N stretching [18-19] because of formation of a complex of molybdenum thiourea from this data I got clear reflection of phase transition. In **Figure 4(d)** there is more absorbance seen in functional group region and one weak peak of –OH group at 3200-3000 cm^{-1} with less absorbance is still there as this sample is washed excessively with ethanol and doubly distilled water and a small peak of carbon dioxide is being detected may it is cause of moisture absorbed by the sample even after the dry process

From this data we cannot give a proper conclusion but it is helpful in a way for the proof of reaction, it means that the reaction is preceded in a good way. Thus a chemical transformation has been taken.

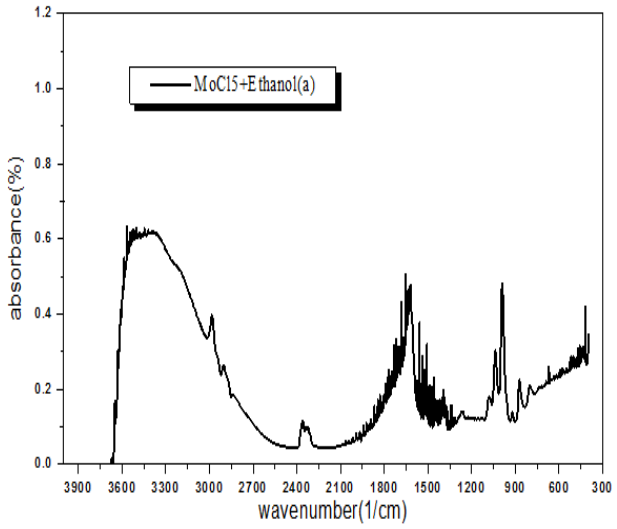

Fig. 4(a): FTIR of $MoCl_5$ in ethanol

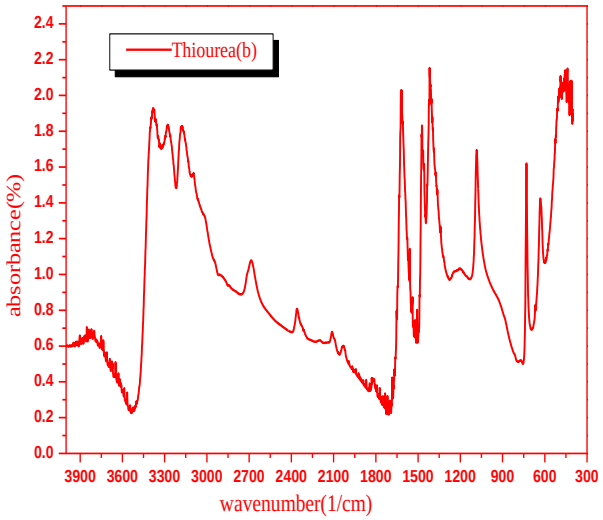

Fig. 4(b): FTIR of pure thiourea

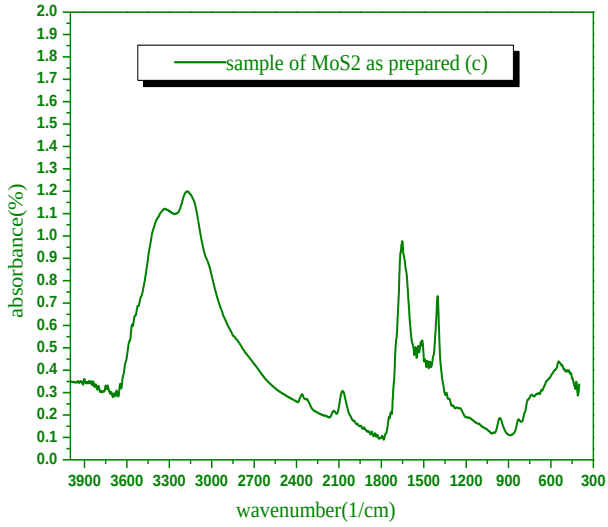

Fig. 4(c): FTIR of dry MoS_2

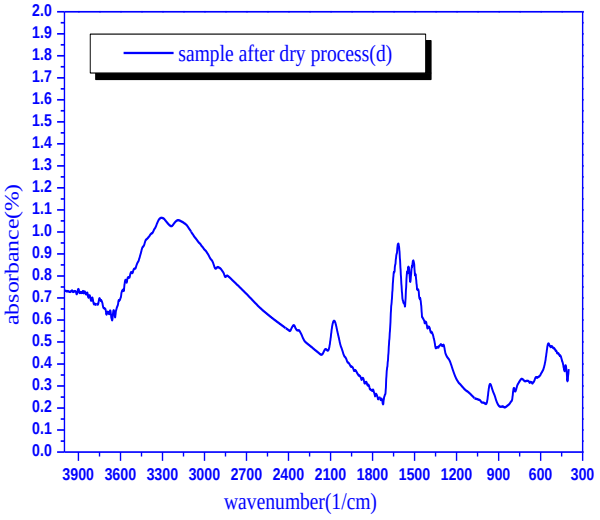

Fig. 4(d): FTIR of as prepared MoS_2

I did preparation of my samples at different parameters in terms of power of microwave oven. Sample(a) at 1400 watt, sample (b) at 1000 watt, sample (c) at 1000 watt (with dry and cleaning process), and sample (d) at 600 watt. Their infra-red data is investigated and interpreted as demonstrated in **Figure (5).** The peak at 250-400 cm^{-1} [10, 19] having strong intensity is assigned to metal (molybdenum)-sulphur stretching vibrations for all the four data below in **Figure (5).** The overtone bands of sample (d) from 1750-1200 cm^{-1}.

It concludes that the reaction of this chemical is yet not completed and of same data is observed for the sample (a) which also indicates the incompletion of reaction. On the contrary, sample (b) and sample (c) gives a flavour of broad and sharp bands not only in finger print but also in functional group region. A clear phase transition can be seen from spectroscopical information of infra- red region. Thus sample (b) and (c**)** gives a complete evidence for reaction mechanism to done in a virtuous manner from this characterisation result.

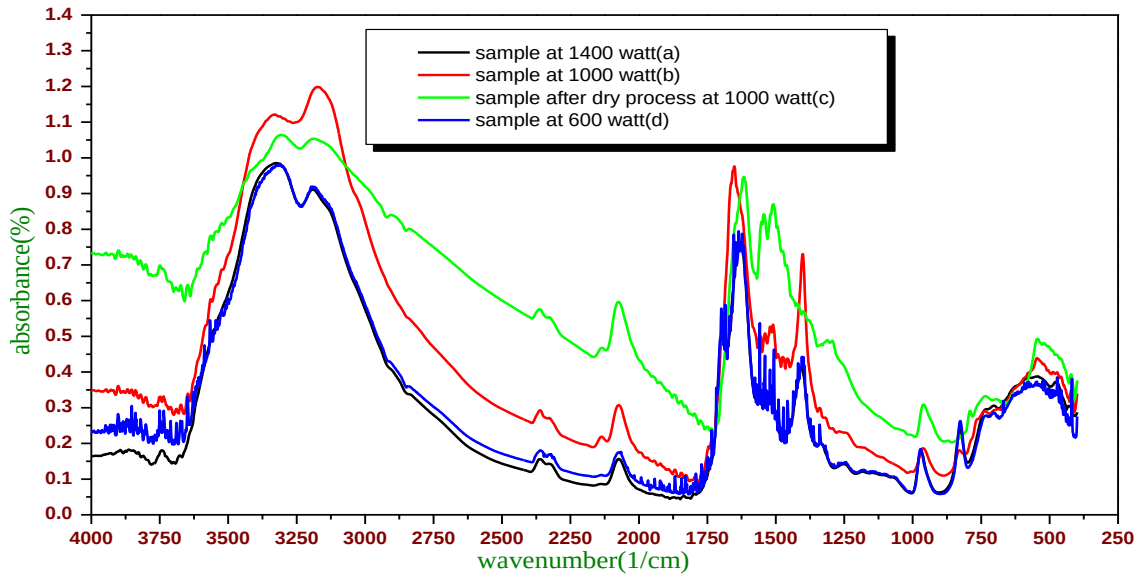

Fig. 5: FTIR spectra of different MoS2 samples prepared at different microwaves powers.

UV-VIS characterization: UV-VIS characterization has been done for the different samples of MoS_2 in NMP, prepared at different concentration of MoS_2 in NMP (i.e., 10 %, 15 % and 25 %). Here, NMP has been chosen as solvent because it surface energy matched with the surface energy of the MoS_2 powder which leads to the exfoliation of MoS_2 multi-layers flakes [20]. It is confirmed from the UV-VIS characterization of the prepared homogeneous mixture of MoS_2 in NMP that for 10 % MoS_2 in NMP there is an absorption peak nearly at 317 nm. While it has been observed that the on increasing the percentage of MoS_2 in NMP, the absorption peak further gets disappear followed by a red shift in the absorption [3].

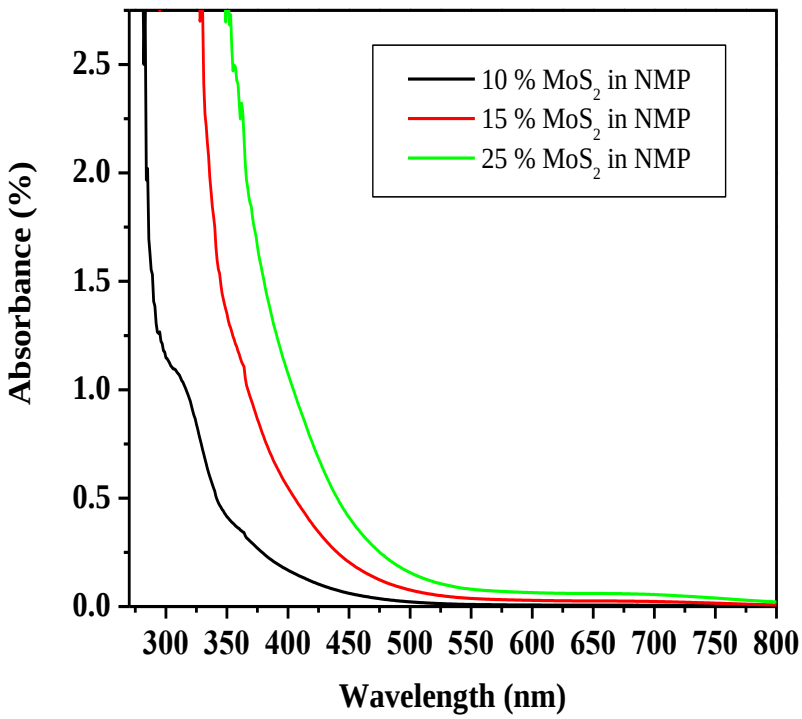

Fig. 6: Show the UV-VIS characterization of different samples of MoS2 in NMP

Scope of study: In this project work, I will prepare composite thin films of molybdenum disulphide and polyvinyl alcohol (MoS_2 – PVA) by using microwave irradiation process and finally, their characterization will be done. The nanocomposite thin films of (MoS_2-PVA) have potential application in the flexible semiconductor electronics (i.e. plastic electronics). Such type of nano-materials can be used to make various electronic components or devices e.g., non-volatile resistive memory, photo sensors, solar cells and batteries etc.

Conclusion: In the present work, a literature survey has been done for the synthesis of the MoS_2 and the study has been done for the various characterizations techniques. After doing the literature survey, I have concluded that synthesis of MoS_2 by microwave irradiation process is a cheap, simple, and less time consuming technique. Hence, in the present work, synthesis of MoS_2 has been done by using microwave irradiation process at different sets of parameters and the FTIR and UV-VIS characterizations have been done for the same. From the FTIR, it is clear that on doing the microwave irradiation of homogenous mixture of $MoCl_5$ + ethanol + thiourea, there is a clear phase transition towards the formation of MoS_2. Furthermore, the UV-VIS spectroscopy confirms that for 10 % MoS_2 in NMP there is an absorption peak nearly at 317 nm and with further increase in the concentration of MoS_2 in NMP there is decrease in absorption peak and a disappearance absorption in the followed by a red shift.

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