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Heterogeneous Mixture of Nanoparticles from MoS_2 and Ta_2O_5: Synthesis and Characterization

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ABSTRACT

The synthesis of metallic nanoparticles is an active area of academic and application research in nanotechnology. It is also an amalgamating technology which has fascinating multi-disciplinary application in various sections. Nanoparticles has been a conventional but field if we go on further decreasing the size we enter the field of quantum dots (<10 nm) with application in the form of tracers, labels, sensors etc. Molybdenum disulfide and tantalum nanoparticles were synthesized and characterized through FESEM, FTIR, XRD,UV-Vis spectroscopy, spectrofluorimetry, etc. The size of the synthesized nanoparticles as observed in FESEM were found to be in the range of 22 to 50 nm for molybdenum nanoparticle and 34.72 to 72.45 nm in case of tantalum. The EDAX analysis shows the composition of molybdenum nanoparticle as hydrogen(H), molybdenum(Mo), nitrogen(N), oxygen(O) and fluorine(F) with 32.3%, 66.3%, 0.43%, 0.32% and 0.5% respectively. The EDAX analysis show the composition of tantalum nanoparticle as hydrogen(H), tantalum(Ta), oxygen(O), nitrogen(N) and fluorine(F) with 35.9%,50%,4.8%,2.01%,7.17%. The XRD analysis of molybdenum disulfide images indicates the synthesized nanoparticle as crystalline in nature. The average crystallinity was found to be 7.93 nm. Tantalum nanoparticles with a crystallinity of 8.05 and 12.20 nm were observed as [2 0 0] and [1 1 0] planes. Biocompatibility of the synthesized nanoparticles was examined by MTT assay. The spectrofluorometry of the synthesized nanoparticles proves the fluorescence property which is most probably because of the quantum dots. Furthermore, the fluorescence property was also used for the cell imaging. The study is a first its kind to exercise the use of Mo and Ta quantum dots in the field of biomedical application and further work is necessary for optimization and implementation of the nanoparticles in the biological sector.

1. Introduction

Synthesis of nanoparticles of varying size and shape has been done using physical, chemical and biological methods. Green synthesis or biological synthesis has been greatly appreciated among all methods, and nano-matters having application in various disciplines such as, physical, medical, optical, material sciences or chemical have been synthesized through this method. For medical usages silver, zinc, cadmium, platinum and gold nanoparticles have been used but inorganic metal nanoparticles and nanoparticles have found uses in colloidal, optical and other physical or chemical uses. Thus the techniques for synthesis of nanoparticles, nanoparticles, uses and properties and characterization methods are reviewed [1-4].

Since the discovery of carbon nano tubes, an entirely new field of scientific study relating to the synthesis and characterization of novel inorganic nanostructures has emerged. Molybdenum disulfide and Tantalum are two of the most promising candidates for creating of novel nano structures due to their unique properties. Molybdenum disulfide can be used as materials for the preparation of electrochemical devices and as a catalyst in catalytic reactions, and it is especially used for lubricants. Tantalum can be used as materials for super conductors, micro batteries, do pants in photo electrode materials. Efficiency of CdTe nano particles was reported [5]. Efficacy of zinc oxide nanoparticle against food borne pathogenic bacteria was reported [6]. Highly efficient dry lubricating film can be formed. Lesser friction coefficient, better catalytic activity and very good physical properties are observed in Molybdenum disulfide nanoparticles. They additionally have a large surface area, high reactivity, and expanded adsorption limit contrasted with the mass material. This black crystalline ore of Molybdenum occurs as the mineral molybdenite. Like graphite, MoS_2 has a low coefficient of friction yet dissimilar to graphite, it does not depend on adsorbed vapors or dampness. MoS_2 coating allows bullets to pass easily through rifle barrels with lower deformation and good ballistic accuracy.

Tantalum is a very rare, blue-gray, hard, lustrous transition metal that is quiet corrosion-resistant. It is a part of the group of refractory metals, which is very widely used as minor components in lot of alloys. The high chemical inertness of tantalum makes it a valuable substance for majority of laboratory equipment and a good substitute for conventional platinum. Its main use today is in capacitors made of tantalum in electronic equipment such as DVD players, video game systems, mobile phones and modern computer systems. Tantalum together with the artificially comparative niobium, happens in the minerals tantalite, columbite and coltan (a blend of columbite and tantalite). Tantalum is a highly conductive material to heat and electricity, and it has made this material a material of choice for electronic capacitors used for hand-held electronic equipment and in telecommunications such as mobile phones and laptops.

The objective of the study is to synthesize molybdenum nanoparticle from molybdenum disulphide powder and tantalum nanoparticle from tantalum oxide powder. Further, the synthesised nanoparticles were undergone physiochemical characterization. The biocompatibility of synthesized molybdenum and tantalum nanoparticles were also evaluated.

2. Experimental Methods

2.1 Synthesis of Molybdenum Nanoparticles

The molybdenum nanoparticles were synthesized using commercial molybdenum disulfide powder. It was dissolved in hydrofluoric acid at a concentration of 400 mg/20 mL. The solution was then stirred on a magnetic stirrer in 700 rpm at room temperature. Randomly add ammonia to the solution. To complete the reaction the mixtures were continuously stirred for 24 hours. The precipitate was separated by centrifugation at

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3000 rpm for 15 min. Then, the supernatant and pellet were collected separately.

2.2 Synthesis of Tantalum Nanoparticles

The tantalum nanoparticles were synthesized using commercial Tantalum oxide powder. It was dissolved in Hydrofluoric acid at a concentration of 400 mg/20 mL. The solutions were then stirred on a magnetic stirrer at 700 rpm. Randomly add ammonia to the solution. To complete the reaction, the mixtures were continuously stirred for 24 hrs. The precipitates were separated by centrifugation at 700 rpm for 15 min. Then the supernatant and pellets were collected separately.

2.3 Characterization of Nanoparticles

The synthesized molybdenum and tantalum nanoparticles have undergone physiochemical characterization like, field emission - scanning electron microscopy (FESEM), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), UV visible spectroscopy, spectrophotometry, Spectro-fluorimetry, Confocal laser electron microscope, cytotoxicity assay (MTT assay) following standard procedures.

2.4 Antioxidative and Nano Enzymatic Activity of Nanoparticles

2.4.1 Peroxidase Mimetic Activity

The peroxidase mimetic action of molybdenum and tantalum nanoparticles were researched by oxidizing the enzymatic substrate TMB within the sight of hydrogen peroxide after oxidation it will give blue shading item and that blue item was then tried. Firstly the experiment was carried out in the absence of hydrogen peroxide and in the presence of nanoparticles mixture. The nanoparticles mixture contains 1 mM TMB (0.9 mL), 1 M acetate buffer (1 mL). The peroxidase activity of nanoparticles (0.1 mg/mL, 20 µL) to oxidize TMB was also investigated in presence of hydrogen peroxide (1 mM, 80 µL). The absorbance change was recorded after 40 min at 652 nm.

2.4.2 Catalase Activity

Teorell-Stenhagen buffer was prepared, which contains 33 mM citric acid, 33 mM phosphoric acid, 23 mM boric acid, at pH = 7.4. Three unique concentrations of nanoparticles were added to the 30 µL of the T-S buffer in presence of 83 mM hydrogen peroxide. After 5 min of response, the solution was then diluted 50 times by using a T-S buffer and the residual hydrogen peroxide was observe in UV-Visible spectrophotometer at 520 nm.

3. Results and Discussion

The physio chemical characterization of Mo and Ta nanoparticles were illustrated in Figs. 1-17. FESEM provided insight into the morphology and details size of the molybdenum and tantalum. The images of FESEM analysis were generated for molybdenum at different resolution and magnification i.e. 200X, 5000X, 10,000X, 3000X, 30,000X (Fig. 1). The size of molybdenum was found to be 100 µm, 5 µm, 1 µm, 5 µm. 50 µm at 200X, 5000X and 10,000X, 3000X, 30,000X respectively. The size of hydrochloric treated molybdenum was 611-821 nm at 5000X and 251 nm - 316 nm at 10,000X (Fig. 2). The reduction in particles size of molybdenum confirms the synthesis of molybdenum nanoparticles. The molybdenum nanoparticles were mostly spherical and rod-shaped. FESEM images of molybdenum also show the presence of the layer-like structure.

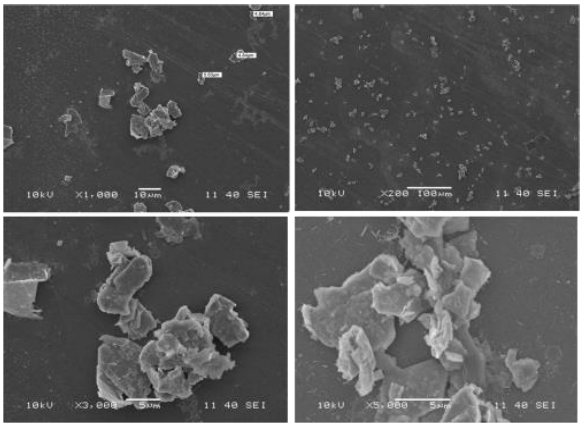

Fig. 1 FESEM image of Mo nanoparticle at a) 1000X b) 200X c) 3000X d)5000X

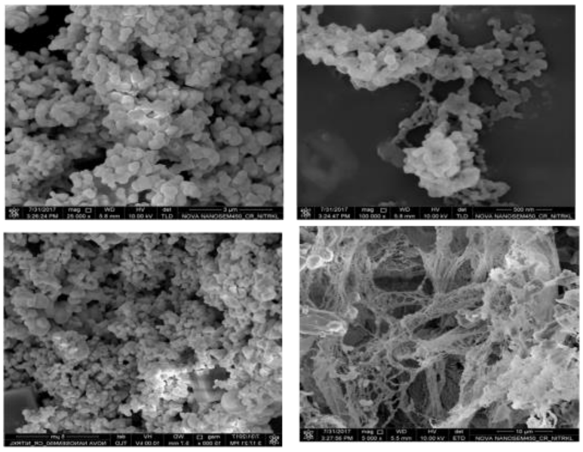

Fig. 2 FESEM image of Ta nanoparticle

The images of FESEM analysis were generated for tantalum at different resolution and magnification i.e. 15000X, 100000X, 25000X, 5000X. The size of tantalum as found to be 100 µm, 5 µm, 11 µm, 5 µm at 15000X, 5000X respectively. FESEM image of Ta nanoparticle sample's surface shows the presence of nanoparticles, which is visible. The aggregated particles can easily be seen in the figure at 10,000X magnification. Again the image reveals the presence of higher number of nanoparticles. Nanoparticles are forming network like structure and they join together. The joining is confirmed by the fibrilous structure. They form spherical like structure also.

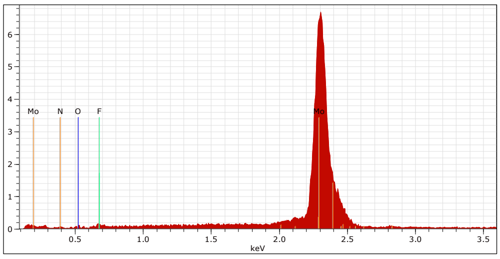

Fig. 3 EDAX of molybdenum disulphide

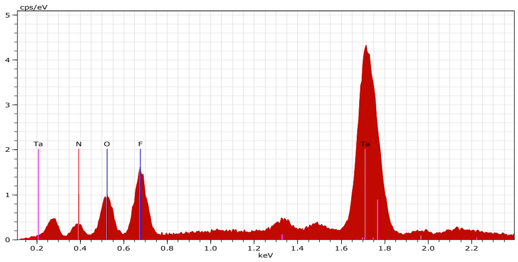

Fig. 4 Elemental analysis of tantalum EDAX (energy dispersive X-ray)

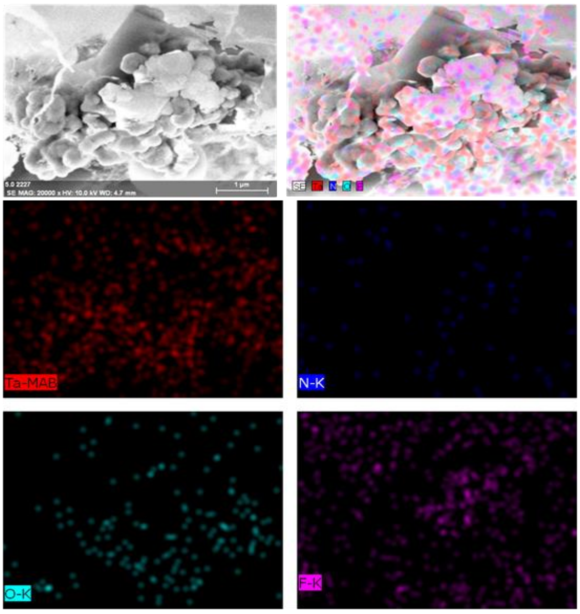

Fig. 5 The structure of tantalum nanoparticles

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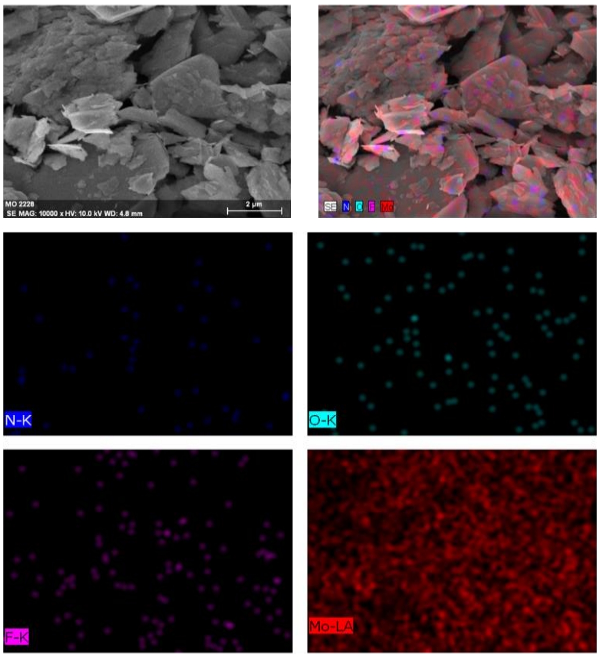

Fig. 6 The structure of molybdenum nanoparticles. The elements such as nitrogen, oxygen, fluorine, and molybdenum are distributed along the whole structure of molybdenum quantum dots

X ray diffraction (Figs. 7 and 8) is a technique used for phase identification of a crystalline material. Generally, the MoS_2 powder is crystalline in nature so the XRD analysis gives different planes like 002, 103 and 105 at different 2ϴ values and the XRD of MoS_2 nanoparticles gives only one plane 002. The different planes like 002, 103, 105 were observed at 2ϴ positions 18°, 47° and 59° respectively. High intensities of these peaks show the 28°.

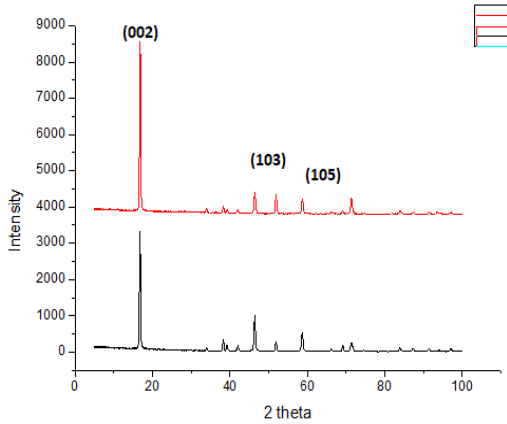

Fig. 7 X-ray diffraction pattern of both MoS_2 and MoS_2 Nanoparticles

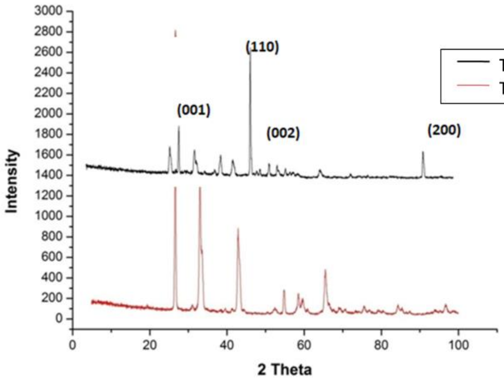

Fig. 8 X-Ray diffraction pattern of Ta_2O_5 and Ta nanoparticles

Spontaneous exfoliation of MoS_2 is achieved in H_2O_2–NMP mixed solvents with a yield of over 60 wt%, operated under mild conditions. H_2O_2-prompted sheet-tailoring effect induces a size evolution of MoS_2 nanosheets from micro-scale to nano-scale. Furthermore, the concurrent dissolution also affords an approach to introduce structural defects in the nanosheets [7]. A study [8] presents a rapid and efficient route to obtain exfoliated 2H MoS_2, which combines fast sonication-assisted lithium intercalation and infrared (IR) laser-induced phase reversion. The results of [9] indicated that, the GO is adhered to the surface of MoS_2 to stabilize MoS_2/GO in solution. Furthermore, this preparation method can also be used to prepare the hybrids of GO and other two-dimensional materials.

Some authors [10] had evolved a scalable and eco-friendly aqueous-based process in combination with renewable and ultra-low-cost lignin which opens up possibilities for large-scale fabrication of MoS_2-based nanocomposites and devices. It has developed two-photon fluorescent (TPF) molybdenum disulfide quantum dots (MoS_2 QDs) through a facile and one-step solvothermal approach. The MoS_2 QDs exhibit small size and high stability. Because of their low toxicity and TPF ability, the MoS_2 QDs are successfully applied in two-photon fluorescence bio-imaging.

QD covering a broad spectrum of interesting optical, catalytic, electronic, chemical and electrochemical properties has been derived and, these 2D-QDs promise a wide range of novel applications including imaging, sensing, cancer therapy, optoelectronics, display, catalysis, and energy [11].

It had been demonstrated that the luminescent molybdenum disulfide (MoS_2) nanosheets, which were prepared hydrothermally by using sodium molybdate and thiourea as precursors, possessed peroxidase-like activity, and could catalyze the oxidation of peroxidase substrate o-phenylenediamine (OPD) in the presence of hydrogen peroxide (H_2O_2) to produce a yellow color reaction [12]. Further addition of Fe^{2+} into the nanosheets led to peroxidase mimetics with greatly enhanced catalytic activity. The observation was exploited to develop a label-free colorimetric nanozyme sensor for detection of Fe^{2+}.

In the XRD analysis of MoS_2 nanoparticles, the intensity of these peaks is lower compared to MoS_2 powder which shows the amorphous nature of the prepared sample. The resulted XRD pattern of the prepared sample ultimately proves the presence of Mo nanoparticles.

XRD analysis is a useful technique that can be used to identify the crystalline structure of samples. Usually, Ta_2O_5 is a crystalline material whose XRD graph gives different crystalline planes like 001, 110, 002, 200 and tantalum nanoparticles are generally amorphous in nature. Different planes like 001, 110, 111, 002, 020, 200 were observed in the XRD pattern of Ta_2O_5 powder.

The FTIR spectrum of molybdenum results the bands at 1409 cm^{-1} bands due to blending vibration of water molecule, 754 cm^{-1} bands due to Mo-O vibration. Fig. 9 shows the FTIR spectrum of tantalum results in the bands at 738 cm^{-1} due to stretching vibration of O-O bond of peroxy group, 483 cm^{-1} due to Ta-O vibration.

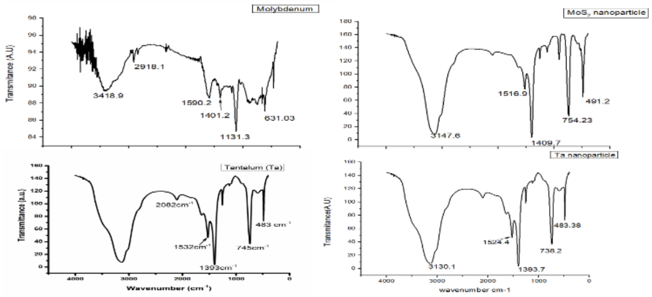

Fig. 9 FTIR spectrum of molybdenum and tantalum

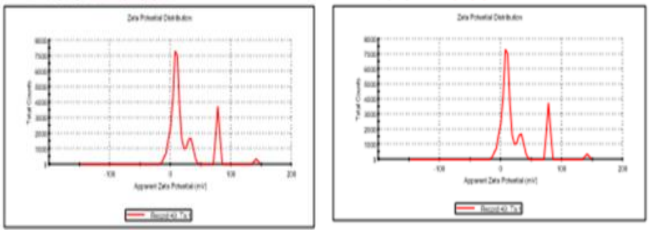

Fig. 10 Zeta potential molybdenum and tantalum nanoparticles

Zeta potential which is the measure for a charge of particles presents on the surface of a compound. The zeta potential analysis which was carried out for molybdenum disulfide nanoparticle for a zeta run of 100 shows that a conductivity of nanoparticle was 6.18 with zero deviation and a zeta potential value of -5.88 as shown in Fig. 10. The zero deviation obtain for particles value depicts the distribution of particle of standard size. The zeta potential analysis which was carried out for tantalum nanoparticle for zeta run of 24 shows that a conductivity of nanoparticle was 1.87 with -2.07 potential with 88.5 deviations.

Confocal was done with Mg 63 cell line. A day before confocal (Figs. 11 and 12) we seeded cell with nanoparticles. And kept for incubation then we took microscopic images. Two major lights were used for confocal i.e. blue and green. At range 400-419 nm blue give fluorescence and 405-488 nm green give florescence. Filter was fixed at 420-460 nm for blue and 500-540 nm for green. After we took the image cells were found florescence under 400 nm. It indicates that the presence of nanoparticle or smaller size particles below 10 nm particles.

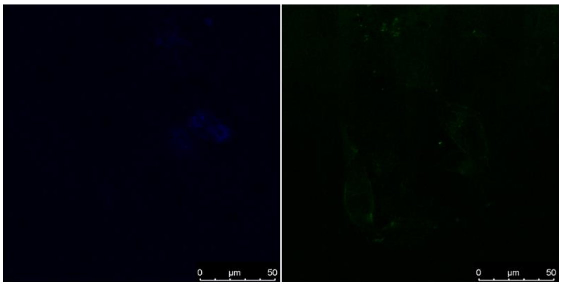

Fig. 11 Confocal laser electron microscopy of tantalum

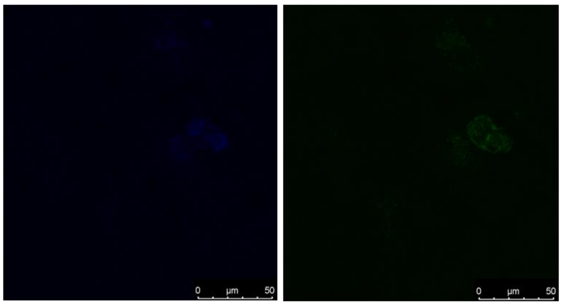

Fig. 12 Confocal laser electron microscopy of molybdenum

Spectrofluorimetry is a technique used to measure the fluorescence spectrum of the sample. Fluorescence spectroscopy of Mo nanoparticles was carried out by varying the excitation wavelength i.e. at 250 nm, 275 nm, 300 nm, 325 nm and 350 nm. As per the Stokes law, the Mo nanoparticle was emitted light at the wavelength of 500 nm, 550 nm, 600nm, 650 nm and 750 nm. Emission of fluorescent light by nanoparticles is a time and size dependent 34 phenomenon and is known to reduce with increase in time and size of the synthesized nanoparticles (Fig. 13).

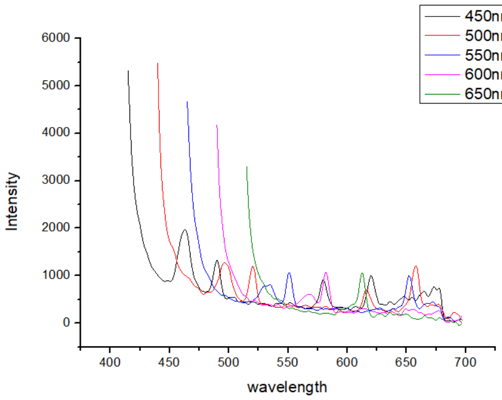

Fig. 13 Fluorescence spectrum of Mo nanoparicle

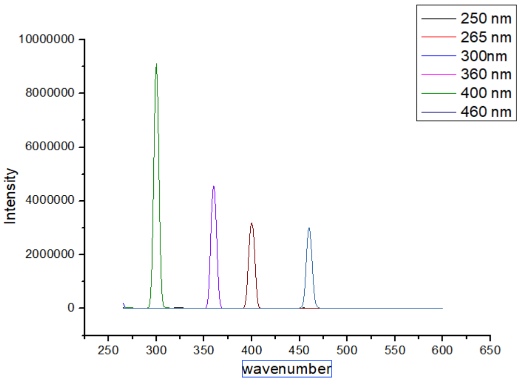

Fig. 14 Fluorescence Spectrum of Ta QD

Fluorescence spectroscopy of Ta nanoparticles (Fig. 14) was carried out by varying the excitation wavelength i.e. at 300 nm, 350 nm, 400 nm and 500 nm. As per the Stokes law, the Ta nanoparticle was emitted light at the wavelength of 610 nm, 705 nm, and 795 nm. Emission of fluorescent light by nanoparticle is a time and size dependent phenomenon and is known to reduce with increase in time and size of the synthesized nanoparticles. The principle of catalase activity is that the catalase should neutralize the toxic form of oxygen metabolites into simpler one, i.e. catalase mediates the breakdown of hydrogen peroxide H_2O_2 into oxygen and water. As we can see that Mos_2 nanoparticles are better catalytic activity than tantalum nanoparticles (Fig. 15).

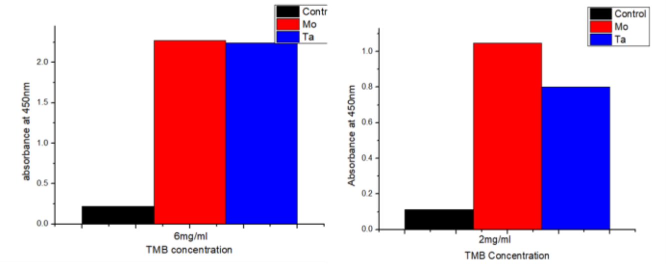

Fig. 15 Catalyse activity of Mo and Ta

The peroxidase like action of metal oxide nanostructures was explored by watching the catalysis of the oxidation of peroxidase substrate, TMB, within the sight of hydrogen peroxide. The dry peroxidase substrate TMB arrangement was gradually oxidized by hydrogen peroxide under acidic condition and it could better and quickly oxidize just in nearness of peroxidases like the action of metal oxide nanostructures like molybdenum disulfide and tantalum nanoparticles and in nearness of H_2O_2 to give a blue color arrangement. It is just because of the charge exchange complex of the parent diamine and the diamine oxidized items. Fig. 16 demonstrating the three diverse TMB arrangement, where the TMB arrangement stay dry by including either nanoparticles or H_2O_2 yet by including both the TMB get oxidized and create the blue shading item. This result indicates that tantalum nanoparticles have peroxidase like activity and can effectively catalyze the oxidation TMB in the presence of H_2O_2 under acetic condition (Fig. 16).

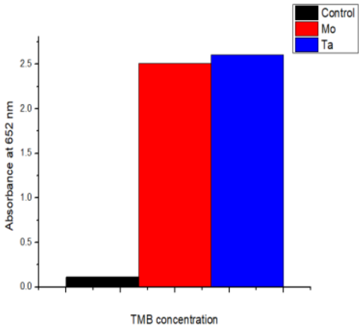

Fig. 16 Peroxidase mimetic activity

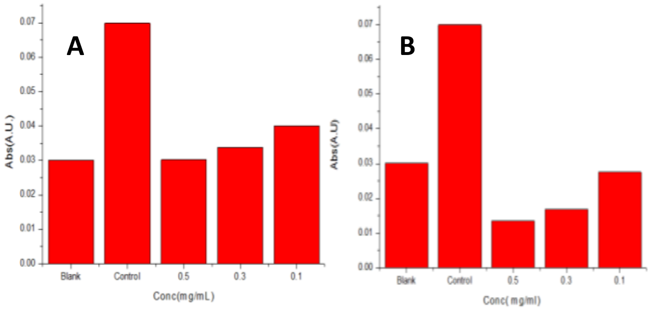

Fig. 17 MTT assay of a) Mo and b) Ta

We tested the peroxidase like activity of metal oxide nanostructures by the TMB concentration i.e. 2 mM TMB and also by varying the concentration of H_2O_2. Tantalum nanoparticles effectively catalyzes the oxidation of enzymatic substrate TMB in the presence 37 of hydrogen peroxide, whereas in case of molybdenum nanoparticles the reaction kinetics decreases.

In-vitro cytotoxicity assay their-vitro cytotoxicity assay of molybdenum disulfide and tantalum nanoparticles in three different concentrations were observed using tissue culture plate (TCP) as a control. The results are

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depicted in Fig. 17. From the graph below it was observed that HeLa cell line (Osteosarcoma bone cancerous cell) show better viability in molybdenum disulfide nanoparticle. By observing the below data we can conclude that good amount of nanoparticles can be used for tissue engineering applications due to enhanced cell proliferation much more than the control (Fig. 17).

The in-vitro cytotoxicity assay of tantalum nanoparticles in three different concentrations was observed using tissue culture plate (TCP) as a control. In 3rd day cell viability index (Fig. 17b) decreases due to longer exposer. By observing the above data we can conclude that we can use a good amount of tantalum nanoparticles for tissue engineering applications due to enhanced cell proliferation much more than the control too. In-vitro cytotoxicity assay of both molybdenum disulfide and tantalum nanoparticles were tested at three different concentrations in the presence of hydrogen peroxide. After adding hydrogen peroxide also the cell viability index increases with an increase in concentration due to antioxidative nature of nanoparticles. From the given data we can conclude that both molybdenum and tantalum having catalytic property due to which metal oxide nanoparticles neutralize the hydrogen peroxide into nontoxic one. Therefore, cell viability index does not hamper by adding hydrogen peroxide into the sample. The cell viability index was found to be higher in comparison to the control used i.e. TCP. Molybdenum disulfide show better result than tantalum nanoparticles.

4. Conclusion

The study on the synthesis of nanoparticles from molybdenum disulfide and tantalum oxide has been used in chemistry, physics and another field of research but its application in the field of biological sciences is restricted. The synthesis of nanoparticles using tantalum, molybdenum disulfide through eco-friendly techniques could boost its application in the field of medical sciences. The heterogeneous mixture of the nanoparticles was formed by treating the MoS_2 and Ta_2O_3 with hydrofluoric acid. Hydrofluoric acids solubilized the MoS_2 and Ta_2O_3 that one addition of ammonia starts nucleating resulted in the formation of nanoparticles. From the FESEM analysis of both molybdenum and tantalum, it is found that there is the reduction of particle size. The spectroflourimetery confirms the presence of molybdenum and tantalum nanoparticles within the heterogeneously distributed particles. The nanoparticles were further characterized by XRD and FTIR. The quantum dots are furthermore used for cell Imaging. The developed quantum dots form Mo and Ta may find further application in various.

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