

DUAL-ROUTE SYNTHESIS OF Nd^{3+} -DOPED MoO_3 NANOSTRUCTURES: UNVEILING PHOTOSENSING AND ANTIOXIDANT POTENTIAL

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ABSTRACT:

Doped nanoparticles of MoO_3 with 1%, 2%, and 3% of Nd^{3+} were prepared at low temperature prepared through sol-gel (powder) or by thin-film (spin coated) method, using the precursor of ammonium heptamolybdate/ $\text{NdCl}_3 \cdot 6\text{H}_2\text{O}$ and characterized for structural, morphological and dual (photosensing–antioxidant) functional properties. The XRD of the 2% composition showed that the sol-gel synthesized material was highly crystalline with an orthorhombic α - MoO_3 phase, while the thin film was less crystalline and presented with a more hexagonal trend without any secondary phases belonging to the Nd_2O_3 system. Both samples contained oxygen vacancies, confirming the presence of Nd^{3+} oxidation states that arose through the substitution of Nd with oxygen vacancies (OVM), as the direct evidence comes from the O 1s spectra with a resolvable oxygen-vacancy component between 531.3–531.6 eV. The Raman spectrum exhibited a terminal $\text{Mo}=\text{O}$ stretching mode redshift to 938–948 cm^{-1} in the sol-gel powder, and a defect-related spectral envelope, which grew with doping level (1855–1962 cm^{-1}) were reported. A defect-related spectral envelope (1855–1962 cm^{-1}), which increased with doping level, was reported along with a redshift of the terminal $\text{Mo}=\text{O}$ stretching mode (to 938–948 cm^{-1}) for the sol-gel powder. FESEM showed that synthesized materials were fine nanofibrous/nanoparticulate where the microplatelet structures present in the materials are seen to coarse grain to irregular agglomerates in the films as the proportion of Nd^{3+} was increased. Functionally, photoresponse increased with doping, higher at 3% Nd (509% (sol-gel) and 617% (thin film)), while concurrently DPPH radical scavenging IC_{50} decreased with doping to 423 $\mu\text{g/mL}$ (sol-gel) and 379 $\mu\text{g/mL}$ (thin film), with the thin film route showing greater effectiveness than the sol-gel powder at all doping concentrations used. The defect-driven and correlated enhancements make 3% Nd-doped MoO_3 thin films the ideal composition—making for a promising mixed-optoelectronic-antioxidant nanomaterial potential in the case of Nd_2O_3 – MoO_3 .

KEYWORDS: Nd_2O_3 -doped MoO_3 ; sol-gel synthesis; thin films; oxygen vacancies; photosensing; antioxidant activity

INTRODUCTION:

The concept of transition metal oxide nanostructures as materials for charge transport is increasingly unusual; the same defect chemistry that controls their charge transport also controls their redox activity in biological media, enabling the same compound to act as a bioactive agent and as an optoelectronic material. A highly interesting example of this can be found in molybdenum trioxide (MoO_3). It is an n-type wide bandgap semiconductor ($E_g = 3.2$ – 3.8 eV) that crystallizes into many morphologies such as nanoribbons, nanosheets, nanowires, nanoflowers, and nanorods with various synthesis pathways [1]. The versatility of this structure makes MoO_3 a recurring material for UV and broad-band photodetection, with MoO_3 interlayers improved the responsivity and detectivity of Si and graphene-based photodiodes [2,5]. MoO_3 -x thin films annealed under inert conditions exhibit nanorod structures which increase the photocurrent output of photodiodes [3]. The defect-engineered MoO_3 nanobelts and core-shell MoO_2 - MoO_3 architectures extend the detection of photocurrent from the UV well into the visible and near-infrared [4,6,7]. All these studies demonstrate that MoO_3 is an oxide with potential Photosensing properties which are very sensitive to morphology, defect density and oxygen stoichiometry, which in principle can be controlled via doping.

Rare-earth doping has become a general solution to that type of control, the substitution of lanthanide ions into host lattices involves substantial lattice distortion and compensating oxygen vacancies, depending on the valence difference with the parent cation, and results in changes of the crystallite size, band gap, and surface defect density in a dose-dependent manner. In similar studies [9–15] of other oxide systems, such as terbium-doped MoO_3 , neodymium-doped CoFe_2O_4 spinel ferrite, and Fe-doped TiO_2 , and Zn-doped Bi_2O_3 , it has been shown that such dopant-induced changes in crystallite size, lattice strain and optical absorption occur as a function of the dopant concentration. The dopant species and the synthetic route are shown to independently influence the final nanostructure, as can be directly concluded from the XRD and FESEM data for MoO_3 nanostructures generated

by the sol-gel method [11]. The same defect chemistry that controls the ability to tune optoelectronic response is also responsible for increasing evidence that MoO₃-based nanostructures have intrinsic antioxidant activity. Synthesized nanoparticles of MoO₃ using co-precipitation and green methods have exhibited measurable DPPH and superoxide anion scavenging capacity, which has been shown to vary according to the precursor acids, the capping agent, and the morphology of the synthesized nanoparticles [16,17,20]. In addition, MoO₃ particles have been demonstrated to possess antioxidant activity in a cell-culture model system, along with dose-dependent cytotoxicity in macrophages [18]; whereas, bio-fabricated MoO₃ nanoparticles possess antioxidant, antibacterial and wound healing activity [19]. The same bioactivity is observed throughout the rare-earth oxide data, with oxygen-vacancy formation and Ce³⁺/Ce⁴⁺ type redox cycling as known mechanisms of superoxide dismutase-mimetic and radical-scavenging properties in cerium, and neodymium, in particular, listed among rare-earth oxides that exhibit measurable reactive-oxidation species-mediated bioactivity [21,22,23]. Doping of a photoactive oxide like MoO₃ with a redox-active lanthanide (such as NdCl₃ – Neodymium Trichloride) is therefore expected to simultaneously alter the defect-driven optoelectronic response, and the radical-scavenging capacity of the oxide, providing a single nanostructured platform with two functionally distinct outputs. Nevertheless, the published literature is largely independent and focuses almost exclusively on either optoelectronic response or antioxidant activity, and there are hardly any reports of both effects being studied in a single doped-MoO₃ system compiled by more than one synthesis procedure.

In the present work, MoO₃ nanostructures doped with 1%, 2%, and 3% of Nd₂O₃ were produced by two independent methods: sol-gel derived powders and spin/dip coated thin films. Elemental composition (EDS), vibrational fingerprinting (Raman spectroscopy), and surface morphology (FESEM) were investigated throughout the entire doping series for both methods of synthesis, with structural evolution probed (XRD and XPS) for the optimized 2% Nd₂O₃ doped MoO₃ composition to establish phase purity and dopant oxidation state. The UV-visible Photosensing ability of the resulting nanostructures was then assessed, followed by their in vitro antioxidant (radical-scavenging) activity to correlate both activities with the dopant concentration and synthesis method, and to prove that the Nd₂O₃-MoO₃ nanostructures can be used as a versatile nanomaterial-platform for various functions.

MATERIALS & METHODOLOGY:

MATERIALS:

The main source of molybdenum (Mo) was ammonium heptamolybdate tetrahydrate (AHM) (NH₄)₆Mo₇O₂₄·4H₂O, ≥99% purity, Sigma-Aldrich/Loba Chemie that was also commonly used as a water-soluble Mo source for the synthesis of hexagonal and orthorhombic MoO₃ [11,20]. The dopant source for neodymium (III) chloride hexahydrate (NdCl₃·6H₂O, ≥99.9% purity, Sigma-Aldrich) which is generally reported as a precursor material with high aqueous solubility and clean thermal decomposition, giving rise to oxide or NdCl₃ species upon calcination [24,25]. Citric acid monohydrate (C₆H₈O₇·H₂O) and ethylene glycol (C₂H₆O₂) were used as chelating and polymerizing agents, respectively, following the Pechini-type sol-gel approach, which promotes homogenous cation distribution and suppresses phase segregation between the host and dopant metal ions during gelation [24]. The precursor solution was acidified to control the pH level and to start the condensation process of polymolybdate, which is a procedure commonly reported for the sol-gel synthesis of hexagonal/orthorhombic MoO₃ nanorods and was performed dropwise with concentrated nitric acid (HNO₃, 69-70%, analytical grade) [11]. A stabilizing/capping agent, polyethylene glycol (PEG-4000/6000) was employed where necessary, following its application for control of particle agglomeration in MoO₃ nanoparticle formation [20]. All synthesis used absolute ethanol (C₂H₅OH, ≥99.9%) and double-distilled/deionized (DI) water as solvents. As typical substrates for metal oxide thin film processing by sol-gel based spin or dip-coating methods, pre-cleaned [glass/FTO/quartz] substrates were employed for the thin film fabrication. All chemicals used were of analytical reagent (AR) grade and were used without further purification, and all aqueous solutions were prepared with DI water with a resistivity of ≥18 MΩ cm.

Synthesis of Nd₂O₃-doped MoO₃ nanopowders (sol-gel route):

Nanorods of pure and Nd₂O₃-doped MoO₃ were prepared by a modified sol-gel process, where a Pechini-type chelation process was used to incorporate Nd³⁺ ions into the ammonium heptamolybdate-nitric acid gelation route reported for hexagonal/orthorhombic MoO₃ nanorods [11,20]. In addition to the undoped sample, several nominal concentrations of NdCl₃ (1%, 2%, and 3%), the ammonium heptamolybdate tetrahydrate was dissolved in deionized water, with the solution continuously stirred by magnetic stirring at room temperature, until a clear and homogenous solution was formed. The molybdate solution was then dropwise added with concentrated nitric acid (69-70%) to reduce the pH and launch the polycondensation process of molybdate species to form a stable solution, following the peptization process reported in AHM-derived MoO₃ gels [11]. The quantities of (NdCl₃·6H₂O) were dissolved in a minimum volume of deionized water and added dropwise to the acidified molybdate solution, following the wet-chemical doping procedures for incorporating NdCl₃ into oxide lattices [24,25]. To ensure a uniform complexation of the dopant cation and to prevent precipitate formation or phase segregation occurring during gelation, citric acid was added to the NdCl₃-containing solution before mixing. The combined solution was then stirred continuously, during which time an increase in viscosity was noted, which indicated the solution to gel transition. The resultant wet gel was then aged to room temperature to ensure completion of network formation like the ambient-temperature aging step observed during the formation of

nanorods of MoO₃ by the sol-gel method. The dried gel was then dried in a hot air oven, resulting in a xerogel. The xerogel was ground to fine powder in an agate mortar and pestle and then calcinated in air with a heating to complete crystallization of Nd₂O₃-doped MoO₃ phase and to burn off the organic residues. The final powders were kept in airtight containers inside the furnace to cool them down to room temperature before characterization was performed.

Fabrication of NdCl₃-doped MoO₃ thin films:

Thin films of pure and Nd₂O₃-doped MoO₃ at different concentrations were deposited on pre-cleaned glass/FTO substrates by the sol-gel spin-coating technique, using the same ammonium heptamolybdate-based precursor chemistry, following the well-established route for depositing α -MoO₃ thin films from AHM-derived solutions [24, 25]. The common use of the same sol-gel precursor system for the growth of the nanopowder and thin film makes it possible to compare then two synthesis procedures under identical chemical conditions and to separate the influence exerted by the different processing routes (bulk gelation/calcination and thin film deposition/annealing) on the resulting nanostructure of Nd₂O₃-MoO₃. Before deposition, glass/FTO substrates were repeatedly ultrasonicated in [detergent solution], acetone, ethanol, and deionized water to remove the contaminants, which followed the standard protocols employed for a sol-gel MoO₃ film deposition [29,30]. The precursor solution for deposition of the thinfilms was prepared by dissolving of (NH₄)₆Mo₇O₂₄·4H₂O in methoxyethanol and stirred continuously at room temperature, following reported precursor formulations for the deposition of MoO₃ films based on AHM-methoxyethanol. The cleaned substrates were spin coated with the precursor solution at 3000 rpm and each layer then dried on a hot plate to evaporate the residual solvent prior to further spin coating. The cycle of coat-dry was performed to obtain the desired film thickness in accordance with the multilayer build-up method described in sol-gel spin-coated MoO₃ films, which it is reported that crystallinity and morphology is strongly dependent on the number of layers [26,27]. Alternatively, substrates were pulled from the solution and dried at room temperature, according to the dip-coating process described for the α -MoO₃ nanorod thin-films [30,31]. To encourage crystallization of the NdCl₃-doped MoO₃ phase and remove any residual organic/solvent content, as deposited films were finally annealed in a muffle furnace in heating air, consistent with annealing regimes reported to drive the amorphous to crystalline transition and nanorod-type morphology development in sol-gel MoO₃ films [33,34].

Characterization Techniques:

X-ray Diffraction (XRD):

The crystal structure and the phase purity of the Nd³⁺-doped MoO₃ samples at the 2% doping level were then investigated by X-ray diffraction (XRD, [instrument model: Rigaku Ultima IV] using Cu-K α radiation (λ = 1.5406 Å) at a scan rate over the 2 θ range, consistent with standard diffractometer configurations used for structural analysis of doped MoO₃ and related sol-gel nanostructures [10, 33]. The crystallite size was calculated from the full width at half maximum (FWHM) of the most intense diffraction peak by Debye-Scherrer equation:

$$D = K\lambda / (\beta \cos \theta)$$

....(1)

Where D is the crystallite size, K is the shape factor (0.9), λ is the X-ray wavelength, β is the FWHM in radians and, θ is the Bragg angle. The incorporation of Nd³⁺ in the host MoO₃ lattice was also quantified in terms of lattice strain and dislocation density, based on crystallite size and strain variation with concentration in doped-oxide nanostructures [11].

X-ray Photoelectron Spectroscopy (XPS):

X-ray photoelectron spectroscopy (XPS) on the Nd₂O₃-doped MoO₃ samples (mean Mo, O, and Nd surface compositions were 2%) was performed with monochromatic Al-K α radiation ($h\nu$ = 1486.6 eV). The binding energies have been charge-corrected to the C 1s peak at 284.6 eV, which corresponds to adventitious C. To distinguish Mo⁶⁺ and any reduced contribution from Mo⁵⁺, associated with oxygen-deficient sites, the Mo 3d spectra were deconvoluted, with emphasis placed on the Mo 3d spin-orbit doublet (Mo 3d_{5/2} and Mo 3d_{3/2}, $\Delta E \approx 3.1$ eV), as has been done for MoO₃ nanostructures [36].

X-ray energy-dispersive Spectroscopy (EDS):

The elemental composition (Mo, O, Nd, and Cl residuals, if any) of all samples for the complete doping series (1%, 2%, and 3% NdCl₃) was obtained by EDS on the FESEM instrument with an accelerating voltage of [kV]. Elemental mapping was also performed to confirm the uniform spatial distribution of the Nd dopant within the MoO₃ matrix, as is common practice in EDS quantification to confirm that the nominal versus actual incorporation of the dopants in the oxide nanostructures is uniform over the spatial domain [21,27].

Raman Spectroscopy:

The Raman spectrum of all samples (1%, 2%, and 3% concentrations) was performed using a Horiba Lab RAM HR, both with a 532 nm excitation laser to avoid sample heating/degradation. The characteristic stretching (Mo=O, Mo-O-Mo) and bending vibrational modes reported for orthorhombic/hexagonal α -MoO₃ were used for spectral assignments and particular attention was paid to the additional oxygen vacancy related mode reported near 1003 cm⁻¹ in defective MoO₃ nanostructure as a spectroscopic marker of dopant-induced lattice disorder [39].

Field-Emission Scanning Electron Microscopy (FESEM):

Surface morphology of all pure and Nd³⁺-doped samples (1%, 2%, and 3% both sol-gel powders and thin films) was studied using FESEM (instrument model, Zeiss Sigma) operating at an accelerating voltage of [kV] with a working distance (mm). To reduce charging effects sample were mounted on carbon tape/conductive stubs and,

as appropriate, sputter coated with layer of [Au/Pt] before imaging. Similar to previously published reports on the morphology-doping correlation in rare-earth doped MoO₃ and related oxide nanorods, nanoplates, and nanoflakes [9,10], the morphology of the resulting nanoparticles was evaluated by tuning the concentration of Nd³⁺ in the precursor solution and by applying two different synthesis routes, i.e., solid-state and hydrothermal methods.

Antioxidant Assay Protocol:

Antioxidant activity of the obtained pure and doped with Nd³⁺-doped MoO₃ nanostructures (1%, 2%, and 3% both synthesis routes) was determined in an in vitro manner using widely used methodology for antioxidant activity assessment of nanomaterials based on the scavenging activity of the free radical 2,2-diphenyl-1-picrylhydrazyl (DPPH). A stock solution of DPPH was freshly prepared in methanol and stored in the dark prior to use. A series of suspensions were prepared by dispersing the NdCl₃-MoO₃ powders/thin-film scrapings into methanol under ultrasonication, which would result in a homogenous suspension. According to the standard DPPH incubation protocol [20] the required amount of DPPH stock solution was mixed with each sample suspension at the concentration indicated and then vortexed followed by incubation in the dark at room temperature to ensure that the reaction is complete. The absorbance of the resultant solution was measured at 517 nm by a UV-Vis spectrophotometer covered with a negative control (DPPH solution without a sample) and a positive control (L-ascorbic acid). The percentage of DPPH radical scavenging (% inhibition) was calculated using the equation:

$$\% \text{ Inhibition} = [(A_{\text{control}} - A_{\text{sample}}) / A_{\text{control}}] \times 100 \quad \dots(2)$$

According to the standard formula used for DPPH based antioxidant evaluation, it is calculated using the following formula: A_{control} is the absorbance of the DPPH solution without sample, and A_{sample} is the absorbance of the DPPH solution with the test sample. A linear regression analysis of the dose-response curve (50% DPPH scavenging vs. concentration) was performed for each doping level and synthesis route, enabling the easy comparison of the antioxidant potency of the all three concentrations of NdCl₃-doped MoO₃ samples.

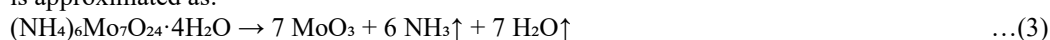
Photosensing Measurement Setup:

The Photosensing (photodetector) function of the pure and Nd³⁺-doped MoO₃ samples, both sol-gel powders (compacted into pellets/electrodes) and thin films, were investigated by the device configuration [heterojunction photodiode] commonly used for UV-visible photodetectors based on MoO₃ [35]. Ohmic/Schottky contacts were fabricated by thermal evaporation, sputtering and silver paste contacts on the sample surface; fine copper and silver conductive paste were used for electrical connections. To determine the rectifying/ohmic properties of fabricated device, a source-measure unit/electrometer (instrument model, Keithley 2400/2450) was used to record current-voltage (I-V) characteristics under dark and illumination conditions at the bias voltage range, with analysis protocol used for MoO₃-based heterojunction photodiodes [34,37]. A UV lamp (365 nm) was used to illuminate the device under test with an optical power density at a specific distance above the surface of the device. In order to analyze the photoresponse of MoO₃-based photodetectors in detail, time dependent photoresponse (I-t) measurements were carried out under periodic ON/OFF illumination cycles with a fixed applied bias of [V] and the following parameters were calculated as the standard figures of merit reported in MoO₃-based photodetectors [37,36]: photocurrent (I_{ph}), dark current (I_{dark}), photosensitivity/on-off ratio (I_{ph}/I_{dark}), responsivity (R=I_{ph}/P.A), and response/recovery time (time taken to reach 90% of the saturation current upon illumination/withdrawal). These parameters were systematically compared among the series 1%, 2%, and 3% Nd-doped sol-gel powder and thin-film samples to determine the effect of the dopant concentration and the synthesis route on the photosensing performance.

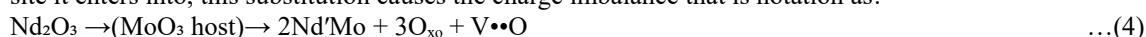
RESULT & DISCUSSION:

Formation mechanism of NdCl₃-MoO₃ nanostructures (sol-gel and thin-film routes):

Both the powder and thin-film methods lead to pure and Nd³⁺-doped MoO₃ nanostructures by the same common sol-gel chemistry. When the (NH₄)₆Mo₇O₂₄·4H₂O is added to water, the results is the release of the ammonium (NH₄⁺) and the Mo₇O₂₄⁶⁻, with it being in a dynamic equilibrium with other isopolymolybdate species as a function of the pH of the solution. Under the action of concentrated HNO₃, the terminal oxo groups are protonated and the condensation reactions known as “olation/oxolation” proceed leading the isolated MoO₆ octahedra into extended Mo-O-Mo chains and sheets who form the sol network [17]. NdCl₃ is added in parallel as NdCl₃·6H₂O dissociates to Nd³⁺ and Cl⁻ with the addition of citric acid, a stable complex of Nd³⁺ and the citric acid is formed, preventing the formation of an early precipitate of Nd(OH)₃/NdOCl and the molecular dispersion of the dopant rather than phase-segregation to the molybdate lattice. The ammonium/organic species undergo a series of stages of collapse as the wet gel is dried to a xerogel and then is heated until it collapses to crystalline MoO₃ at ~500–650 K, which is approximated as:



During the process, the citrate ligand is oxidized to CO₂/H₂O and chloride is volatilized as HCL/NO_x leaving a lattice anchored Nd³⁺. Since the size and charge of Nd³⁺ (~0.983 Å) are very different from the Mo⁶⁺ (~0.59 Å) site it enters into, this substitution causes the charge imbalance that is notation as:



Which is also consistent with the physiologically relevant oxygen-vacancy mediated effects on antioxidant and antibacterial activity, is also responsible for the changes in crystallite size, strain, and defect states as a function of concentration. The gel formation process between the two synthesis routes is chemically identical, and a only distinct difference is the final thermal step: a slow, uniform furnace ramp profile (powder route) generally results

in more complete crystallization and larger more agglomerated grains, while a thin layer of high surface area gel (thin film route) typically leads to more rapid loss of solvent/organic material volume and heterogenous nucleation of the interface having more rod/plate-like structures at the same nominal annealing temperature [32,34,37]. The morphological contrast and the crystallite size contrast between the sample series described in the XRD and FESEM sections would be the main origins of this processing-route difference.

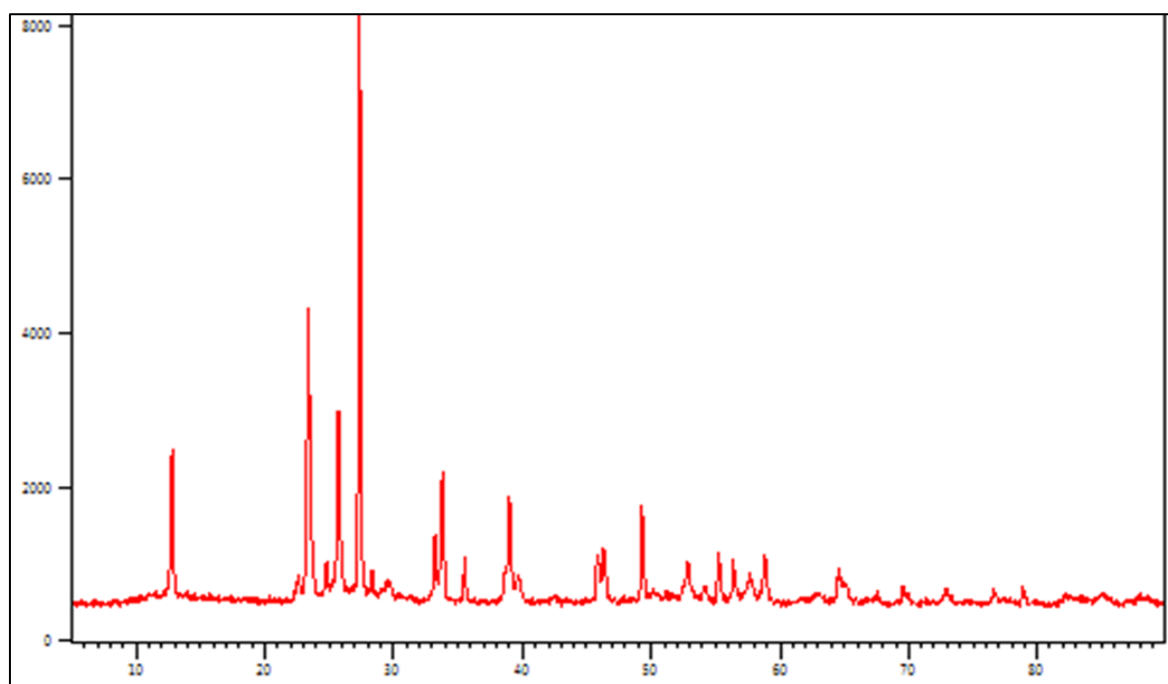
XRD Analysis:

The XRD patterns of the 2% Nd₂O₃-doped MoO₃ samples prepared by the sol-gel (MN-2%) and thin-film (TF-2) routes are displayed in Figure 2(a) and 2(b) respectively and indicate huge difference in the phase development and crystallinity of the two methods, despite the same precursor chemistry. The sol-gel powder (MN-2) exhibits a sharp, well-resolved multi-peak diffractogram with diffraction maxima at approximately $2\theta \approx 12.6^\circ, 23.3^\circ, 25.7^\circ, 27.3^\circ, 33.7^\circ, 38.9^\circ, 39.6^\circ, 46.2^\circ, 49.2^\circ, 53.1^\circ, 58.7^\circ$, and 64.5° , superimposed on a low, flat background. This peak sequence — and in particular the characteristic triplet in the $23\text{--}28^\circ$ region with the (021) reflection near 27.3° dominating in intensity — is diagnostic of the orthorhombic α -MoO₃ phase (space group Pbnm, JCPDS Card No. 05-0508), and can be indexed to the (020), (110), (040), (021), (111), (060), (150), (002), (211), and (081) planes, respectively. No secondary phase reflections of Nd₂O₃, NdOCl, or NdMoO₄ are detected up to 2% suggesting that no dopant phase is formed, but rather than Nd³⁺ is incorporated into the MoO₃ framework, most likely into Mo⁶⁺ sites, consistent with the substitutional defect mechanism. The sharp high-intensity and low background profile of MN-2 is evidence of high crystallinity, as in the powder route the specimen can be available for slow and uniform heat treatment.

The diffractogram for the thin-film sample (TF-2) is characterized by a broad, high background that is accompanied by a few discrete, comparatively weak features, a strong peak at low angle (near $2\theta \sim 8\text{--}9^\circ$), a weaker peak at $\sim 23\text{--}24^\circ$, a third weaker peak at $\sim 33^\circ$, and a moderate intensity peak at $\sim 48\text{--}49^\circ$. The underlying broad hump is due to the amorphous glass/FTO and a remaining amorphous/nanocrystalline film contribution, and the intense low-angle reflection corresponds to the basal reflection of the (100) crystal planes of the layered, metastable hexagonal h-MoO₃ Phase (JCPDS Card No. 21-0569) that appears in this low-angle region, whereas the orthorhombic α -MoO₃ fingerprint is present for MN-2. This phase divergence is route-dependent, resulting from rapid, substrate nucleated annealing that does not afford the same thermal budget as bulk furnace calcination, facilitating the easier nucleation of the kinetically favored hexagonal polymorph and leaving a higher number of amorphous structures in the film than would bulk furnace calcination, which would drive the film completely into the thermodynamically stable orthorhombic phase [28,30].

Overall, the XRD results confirm that there are no evidence of secondary phases of the dopant or its compounds in the (Nd)MoO₃ produces at 2% Nd, while the particular heat history of the two synthesis routes gives the resulting products different polymorphs and crystallinity: orthorhombic α -MoO₃ for the sol-gel powder and a less crystalline hexagonal-leaning polymorph for the thinfilm.

(a)



(b)

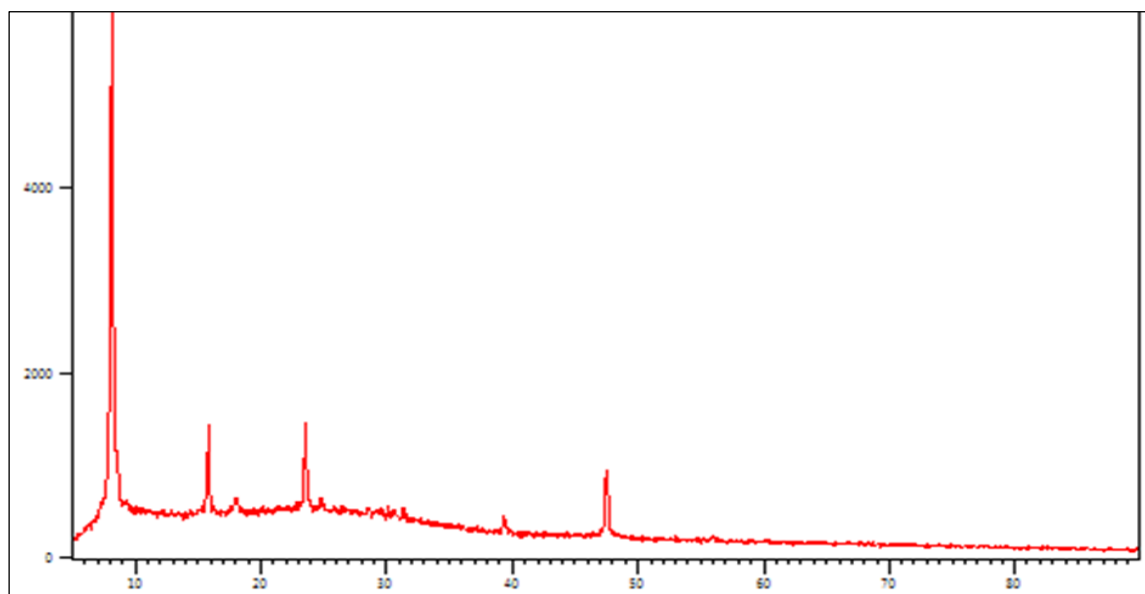


Figure 2: XRD Analysis of (a) Sol-gel route (2% Nd_2O_3 -doped MoO_3) & (b) Thin-film route (2% Nd_2O_3 -doped MoO_3)

EDS Analysis:

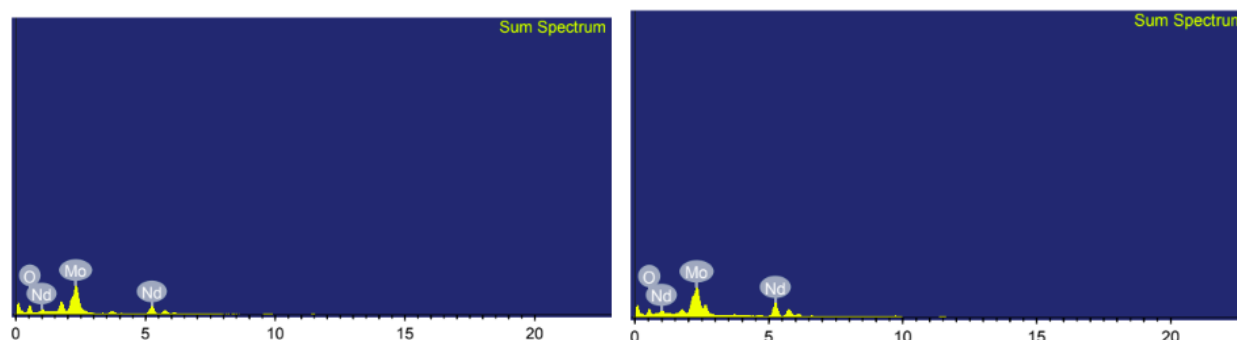
The elemental composition of the prepared samples of Nd^{3+} - MoO_3 at all the doping levels (1%, 2%, and 3%) for the sol-gel powders and thin films was investigated using EDS spectroscopy and elemental mapping, and representative EDS spectra and elemental maps are shown in Figure 3 & 4, respectively. The EDS spectra are free from any detectable peaks of chlorine, nitrogen or carbon; for both synthesis routes and at all doping concentrations only three elemental signals are seen, namely O $\text{K}\alpha$, Mo $\text{L}\alpha$ and Nd $\text{L}\alpha$. The lack of the Cl residue confirms that the volatilization of Cl is complete during with the drying/calcination (powder) and annealing (film) steps, as discussed with the formation-mechanism. For both preparation methods, the indicates that Nd is only found in the final product in an oxide-coordinated (lattice-substituted) form, and not in the form of a residual chloride phase.

The intensity of the Nd $\text{L}\alpha$ signal relative to the prominent Mo $\text{L}\alpha$ signal exhibits a clear trend with increasing nominal Nd concentration in the 1% to 3% samples, both in the sol-gel and thin-film series, and points to the progressive from the structure or its loss during processing. The corresponding maps of the elements (O $\text{K}\alpha$ 1, Mo $\text{L}\alpha$ 1, Nd $\text{L}\alpha$ 1) reveal a uniform distribution of oxygen and molybdenum across the area imaged for all samples, which represents a continuous oxide matrix, and a spatially uniform dopant distribution (Nd $\text{L}\alpha$ 1) without segregation picture of $\text{Nd}_2\text{O}_3/\text{NdOCl}$. Although the difference is subtle, there is a clear difference between the two routes: maps of the sol-gel powders exhibit a fine, evenly speckled distribution, while the maps of thin films show a somewhat more clustered/patchy texture with a broader, less crystalline XRD profile being obtained for the thin-film route (TF-2) compared to MN-2.

Overall, the EDS data confirms that both the sol-gel and the thin-films samples are phase-pure, containing no detectable amount of chlorine or other impurity elements; that the amount of Nd has monotonically increased with dose for both of the routes; and that the distribution of Nd is spatially homogenous in both samples, as also is the distribution of elemental texture, with the latter being more uniform in the sol-gel powders than in the thin films.

(a)

(b)



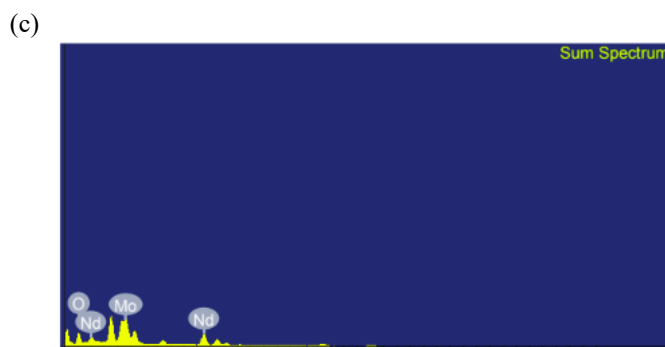


Figure 3: EDS of Sol-gel route Nd_2O_3 -doped MoO_3 nanostructures (a) 1%, (b) 2%, and (c) 3%

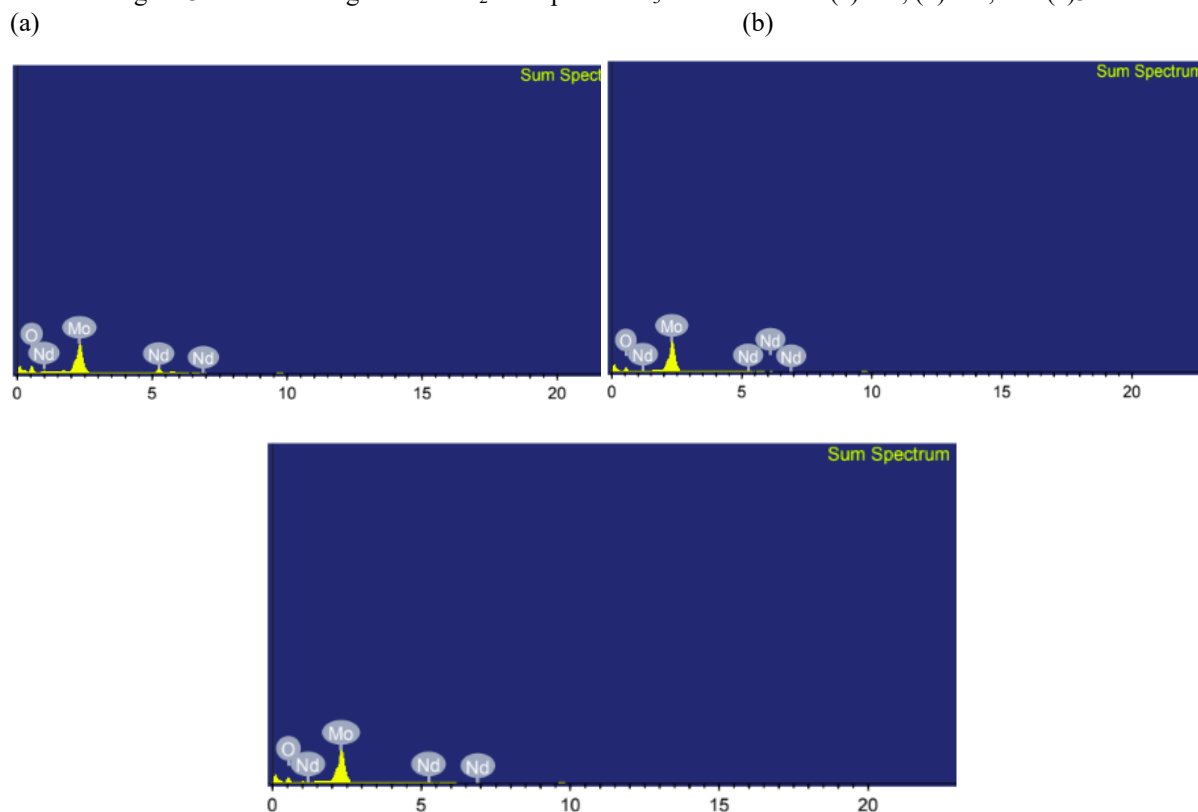


Figure 4: EDS of Thin-film route Nd_2O_3 -doped MoO_3 nanostructures (a) 1%, (b) 2%, and (c) 3%

XPS Analysis:

XPS was used to investigate the chemical composition and oxidation state of the 2% Nd_2O_3 -doped MoO_3 prepared via the sol-gel (Figure. 5a–d) and thin-film (Figure. 6e–h) methods using the adventitious C 1s peak at 284.8 eV as a reference. The survey spectra presented in Fig. 5a and 5e confirm the presence of O, Mo and Nd, the former two elements being the only major component in both samples, and the presence of C 1s signal, which is almost always found with both samples, indicating adventitious carbon; no signal from chloride is detected in either of the survey scans, both of which compare well to the EDS result that no chloride is present after thermal processing except for that in the $\text{NdCl}_3 \cdot 6\text{H}_2\text{O}$ precursor, and that it is greatly reduced and incorporated in an oxide coordinated form.

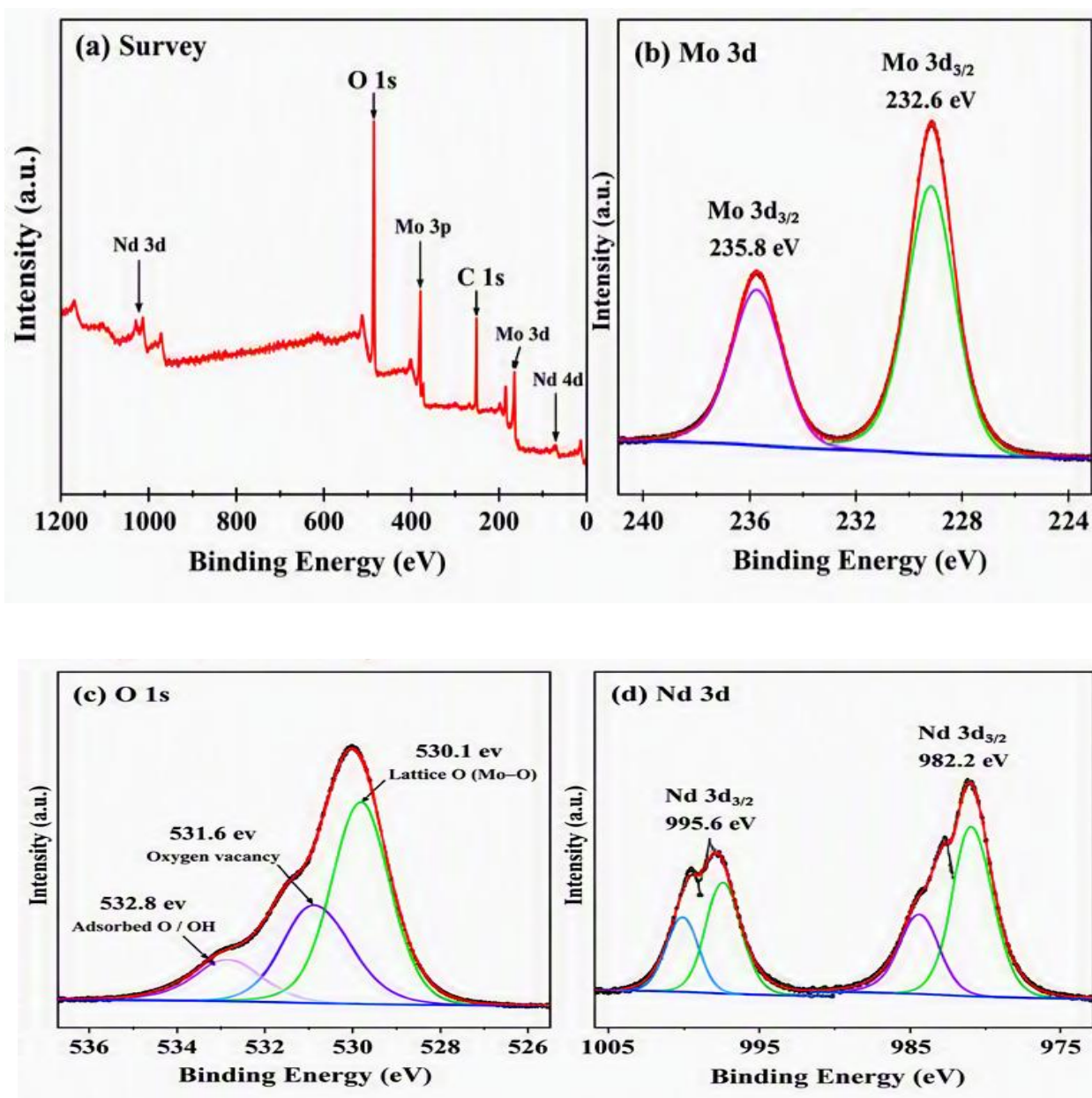
Mo 3d region, the spectra of both samples shows one reasonably well-resolved Mo 3d spin-orbit splitting, where Mo $3d_{5/2}$ occurs at 232.6 eV and Mo $3d_{3/2}$ at 235.8 eV for the sol-gel sample, and Mo $3d_{5/2}$ at 232.3 eV and Mo $3d_{3/2}$ at 235.6 eV for the thin film sample (Fig. 5b, f). In both, the spin-orbit splitting (≈ 3.2 – 3.3 eV) and the position of the binding energy is representative of Mo^{6+} in an octahedrally coordinated MoO_3 environment, with no distinct Mo^{5+} (reduced) feature being resolved on the lower binding energy shoulder. This suggests that the Mo oxidation state is largely Mo^{6+} , even at the surface sensitive probing depth of XPS, for both routes, and that the charge compensation needed by Nd^{3+} substitution is accomplished primarily by the creation of oxygen vacancies rather than by the reduction of Mo^{6+} to Mo^{5+} . This lends credence to the vacancy-dominated defect picture proposed in this work, rather than a mixed-valence Mo picture.

O 1s region, the XPS O 1s spectra (Fig. 5c, g) shows that the spectra can be deconvoluted into three peaks for both routes: a major peak at 530.1 eV (sol-gel) / 529.9 eV (thin film) was attributed to the lattice oxygen (Mo–O) species; an intermediate peak at 531.6 eV (sol-gel) / 531.3 eV (thin film) is attributed to the oxygen-vacancy-associated oxygen species; a minor peak at 532.8 eV (sol-gel) / 532.6 eV (thin film) is attributed to surface-

adsorbed oxygen/hydroxyl species. The clear identification of a distinct oxygen-vacancy component in both samples provides direct XPS evidence for the presence of a vacancy-sensitive component of defects as predicted by the Nd^{3+} -substitution mechanism, which also supports chemical evidence of such defects from the Raman defect envelope observed and the redox-active surface implicated in the antioxidant activity.

Nd 3d region, for both samples there is a well-defined Nd 3d doublet, with the Nd $3d_{5/2}$ component at 982.2 eV and the Nd $3d_{3/2}$ component at 995.6 eV in the sol-gel sample, and 981.8 eV/995.2 eV in the thin film (Fig. 5d, h), along with the characteristic Nd 3d multiple splitting satellite structure (the additional lower intensity component adjacent to each main peak), typical of Nd^{3+} . Both routes give rise to binding energies consistent with neodymium being incorporated in the trivalent (Nd^{3+}) oxidation state; as discussed in, there is no evidence for any higher oxidation state of Nd.

Route comparison, the binding energy of all three core-levels (Mo 3d, O 1s, Nd 3d) in the thin film show a small but consistent negative shift (~ 0.3 - 0.4 eV) compared to the sol-gel powder. This is not a charge-correction artifact, but a genuine very small difference in the local electron density around Mo, O and Nd, between that of the more fully crystallized, stoichiometric sol-gel powder samples and the less crystallized, more amorphous/defective thin-film samples. However, the overall results from the XPS analyses indicated that both synthesis pathways gave MXPO oxidation states that are chemically equivalent, with a similar surface with oxygen vacancies at 2%, and that the differences in the position of the binding energies of the elemental components and thus of the defect environment are rather minor, and dependent more on the synthesis than on any other factor.



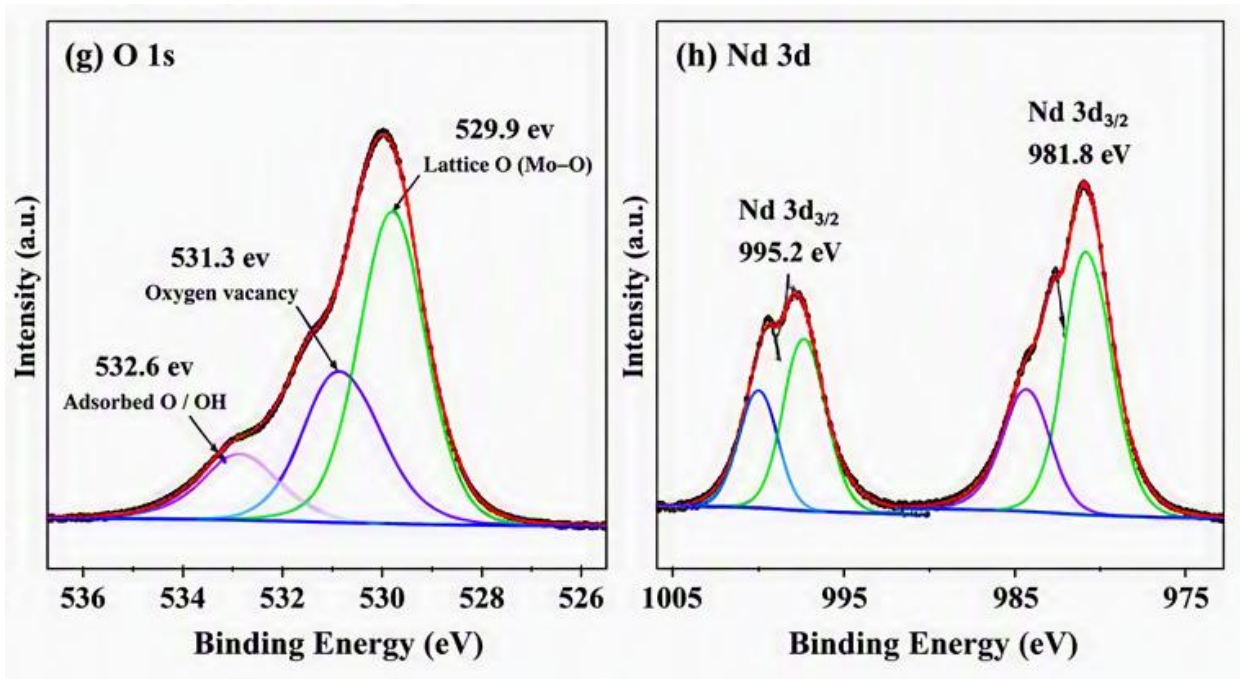
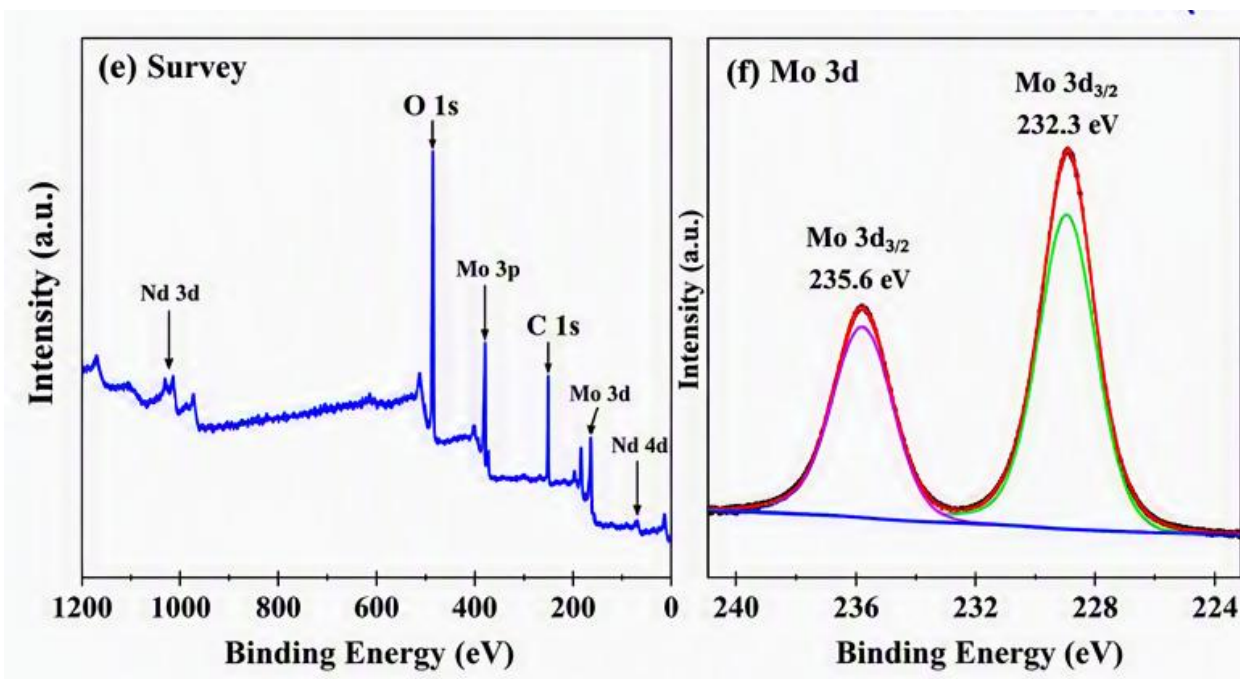


Figure 5: XPS analysis of Sol-gel route (2% Nd_2O_3 -doped MoO_3) [a-d] & Thin-film route (2% Nd_2O_3 -doped MoO_3) [e-h].



MoO_3 [e-h]

FESEM Analysis:

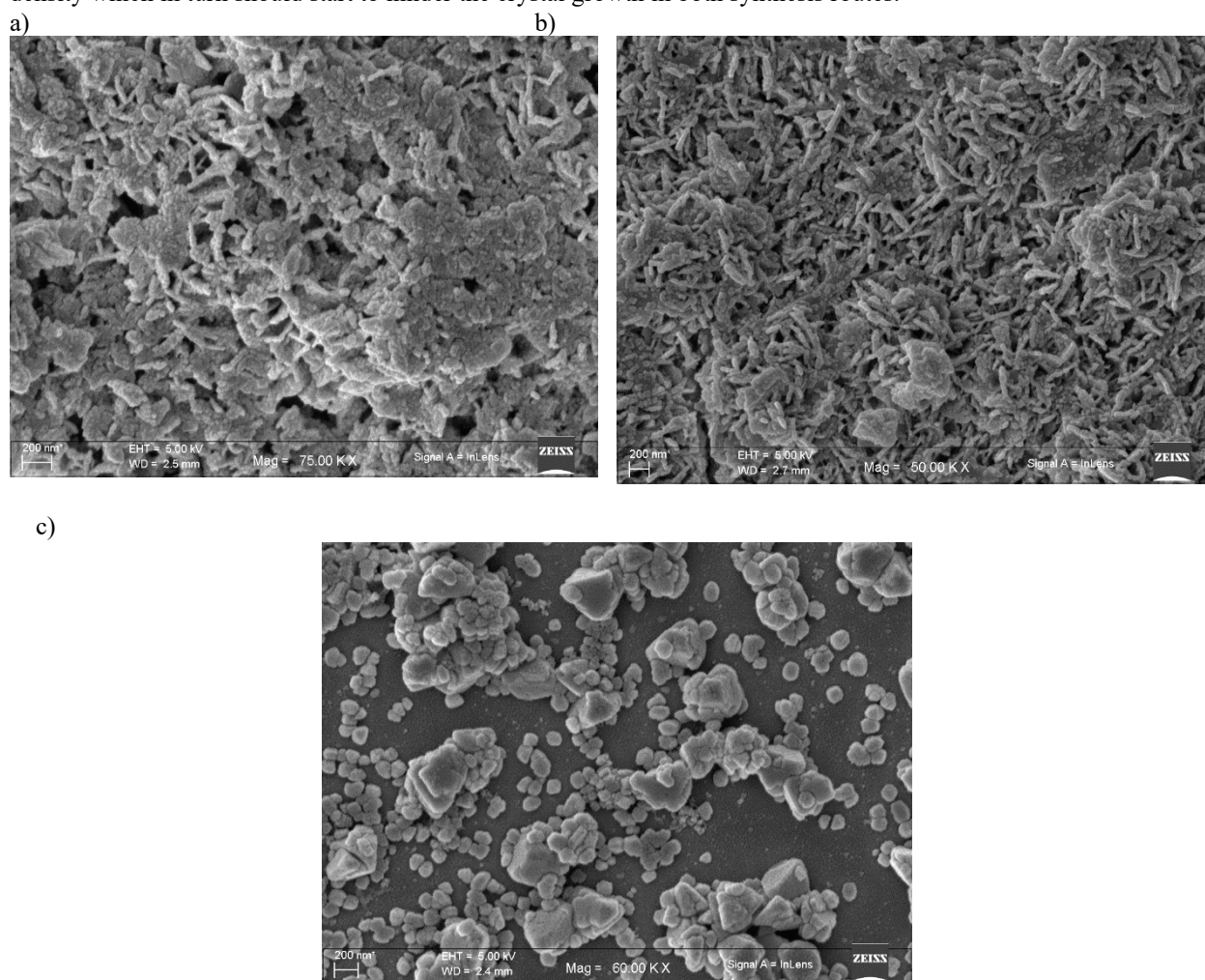
The surface morphology of the obtained Nd^{3+} -doped MoO_3 samples with doping concentration of 1%, 2%, and 3% prepared by both the sol-gel and thin-film routes is presented in Figure. 6 (a-c) [sol-gel] & Figure. 6 (d-f) [thin-film], as a clear morphological evolution is seen as a function of doping concentration and is different for the two routes of synthesis.

Sol-gel powders show a dense agglomerated fine-grained morphology with irregular clusters of nanoparticulate growth mode (less than 100 nm) and some short, needle-like fragments of nanorods (NRs) (nucleation-dominated growth), which is typical of the low-content of the dopant in bulk xerogel calcination. As the amount of Nd^{3+} is increased to 2% (MN-2), the morphology changes to a continuous, fibrous network consisting of nanorods and nanofibers which cover the entire surface of the sample, suggesting that an optimum concentration of Nd^{3+} has shifted towards anisotropic, one-dimensional crystallite growth opposite to that observed in the case of 1% (MN-1). At 3% (MN-3), the morphology changes once more to well-faceted, polygonal (triangular/hexagonal-plate) microcrystallites, with a low weight fraction of very fine particulates (sub-100 nm), which form loose aggregates without strong correlations in terms of size or shape. This sequence from the fine agglomerated grains to the

fibrous network of nanorods to larger faceted crystallites, goes from promoting elongation in the direction of the electric field, to driving coarsening of the structure into discrete crystallites by Ostwald-ripening like growth, possibly assisted by defect chemistry induced by higher substitution level of Nd^{3+} into the FeO layer.

In the thin-film series a completely different and (at low doping) much more ordered morphology is observed. For the 1% film (TF-1) a dense array of large well faceted elongated rod/plate-like crystallites is found, covering the substrate nearly uniformly, and characteristic of highly crystalline orthorhombic $\alpha\text{-MoO}_3$ has the growth habit of a microplatelet. In this rod/plate array in a 2% TF-2, although the primary, well defined morphology of the plates still exists, it is accompanied by other regions of finer, granular agglomerated material, which represents a secondary growth mode. By 3%, the sharp rod/plate-like shape is largely replaced by coarse granular aggregates containing occasional resplendently rod shape fragments which suggests that the oriented growth into microplatelets from the undoped/lightly doped film at the same grade is strongly influenced by higher loading of Nd^{3+} .

The particles after coming out of the sol-gel powders at the same nominal doping levels always come out at smaller, more nanoscale dimension (nanoparticles/nanofibers versus micron-sized crystallites of rods or plates) than the particles formed in thin films (micron crystallization sizes) when doped at low levels. This is consistent with the different thermal/growth environments postulated in the bulk powder case more nucleation sites and limited grain growth are sought during furnace calcination while in the substrate-confined thin-film case fewer, larger, more oriented crystallites are desired during annealing; this latter desire would be overturned, however, by the increased lattice strain and oxygen-vacancy disorder caused by higher contents of Nd^{3+} (2–3%), resulting in more agglomerated and less-ordered morphologies at the highest doping level in both cases. This common high-doping behavior (higher agglomeration/irregularity at 3% in both the routes) also supports the proposed defect-compensation mechanism described earlier, in which excess Nd^{3+} substitution leads to a higher oxygen-vacancy density which in turn should start to hinder the crystal growth in both synthesis routes.



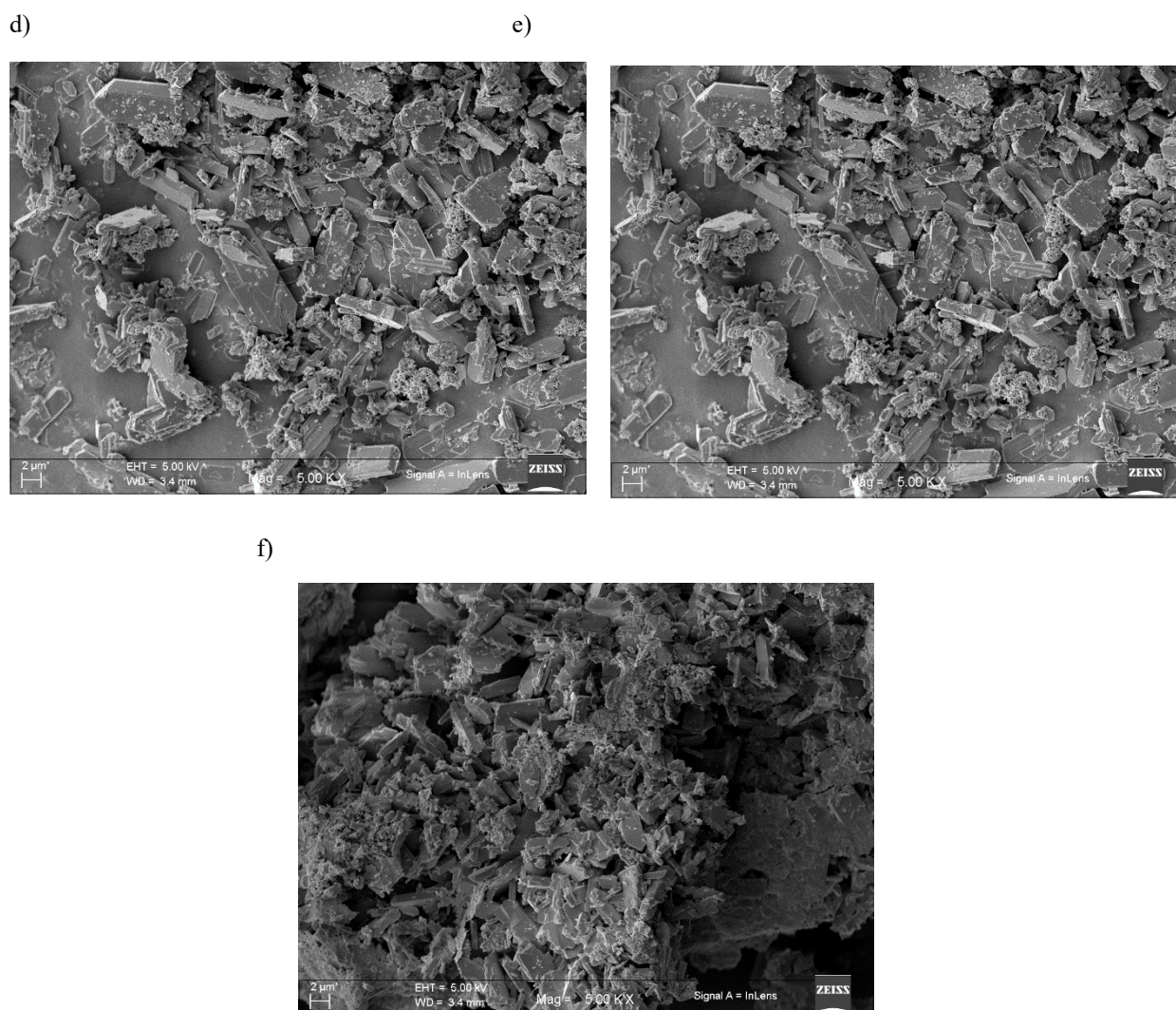


Figure 6: FESEM Analysis of Sol-gel route (Nd_2O_3 -doped MoO_3) a) 1%, b) 2%, c) 3% & Thin-film route (2% Nd_2O_3 -doped MoO_3) d) 1%, e) 2%, f) 3%

Raman Analysis:

Both syntheses reproduce the characteristic lattice ($\sim 77\text{--}93\text{ cm}^{-1}$) and bending ($\sim 329\text{--}342\text{ cm}^{-1}$) modes of orthorhombic $\alpha\text{-MoO}_3$, which is established for the sol-gel powders. In both series, a slight softening of both modes is seen as the concentration of Nd^{3+} grows, which is the sign of progressive distortion of the lattice, as is expected following progressive Nd^{3+} -for- Mo^{6+} substitution and oxygen-vacancy compensation proposed mechanism (Table 1 and Figure. 7).

The most obvious distinction which depends on the route is the stretching mode of the terminal $\nu(\text{Mo}=\text{O})$. This mode is in the ordered, near-stoichiometric $\alpha\text{-MoO}_3$ ($\sim 992\text{--}996\text{ cm}^{-1}$) range for the thin films and changes only slightly ($\Delta \approx 3.5\text{ cm}^{-1}$) over the entire doping range. The by comparison sol-gel powders, however, have a much greater deviation from the ideal terminal oxygen bond, evident as a very significant red shift to $\sim 938\text{--}948\text{ cm}^{-1}$. Combined with the FESEM and XRD this demonstrates that in the sol-gel powders, more Nd^{3+} ions occupy well-ordered lattice sites (directly weakening the terminal $\text{Mo}=\text{O}$ bond) while in the thin films with a larger amorphous/less-crystalline content, a larger percentage of Nd^{3+} may occupy grain boundaries, interfacial, and amorphous regions, and therefore less affect the terminal-oxygen bond environment of the crystalline component. Both routes exhibit a very similar and regular growth of a broad envelope around $1850\text{--}1950\text{ cm}^{-1}$, whose location is accompanied by the redshift with increasing Nd^{3+} content in both series (sol-gel: $1854 \rightarrow 1898 \rightarrow 1962\text{ cm}^{-1}$; thin film: $1855 \rightarrow 1898 \rightarrow 1951\text{ cm}^{-1}$). The consistently observed growth of this feature with increasing oxygen-vacancy density after different Nd^{3+} doses on both independently-processed sample sets is a valuable cross-validating signature of increasing oxygen-vacancy density upon increasing Nd^{3+} doping, despite its high Raman shift for a fundamental MoO_3 vibration, even though it is attributed to a broad, defect/oxygen-vacancy mediated photoluminescence-type background; directly corroborated it the defect-compensation mechanism, and prepared the ground for the discussion of oxygen-vacancy-mediated antioxidants.

Mode Region	Route	1%	2%	3%	Assignment
Lattice Mode	Sol-gel	92.8	77.6	81.9	Ag rigid-layer/lattice vibration
	Thin-film	83.2	82.1	81.0	Ag lattice vibration
Bending Mode	Sol-gel	34.18	330.8	328.9	$\delta(\text{O}=\text{Mo}=\text{O})$ bending
	Thin-film	332.4	330.2	328.8	
O-Mo-O bending	Thin-film	667.1	665.4	663.2	O-Mo-O deformation
Bridging Stretch	Sol-gel	822.9	875.7	824.6	$\nu(\text{Mo}-\text{O}-\text{Mo})$
	Thin-film	821.6	819.8	817.4	
Terminal Stretch	Sol-gel	870.7/939.8	948.2	938.1	$\nu(\text{Mo}=\text{O})$
	Thin-film	995.8	994.1	992.3	
Combination band	Sol-gel	1096.2	1095.6	-	Overtone/combination
	Thin-film	1096.4	1093.8	1090.5	
Defect envelope center	Sol-gel	1853.8	~1898	~1962 (avg.)	Oxygen-vacancy/defect-related band
	Thin-film	1855.2	1897.6	1951.4	

Table 1: Fitted Raman mode positions (cm^{-1}) for Nd_2O_3 - MoO_3 nanostructures as a function of Nd^{3+} doping and synthesis route

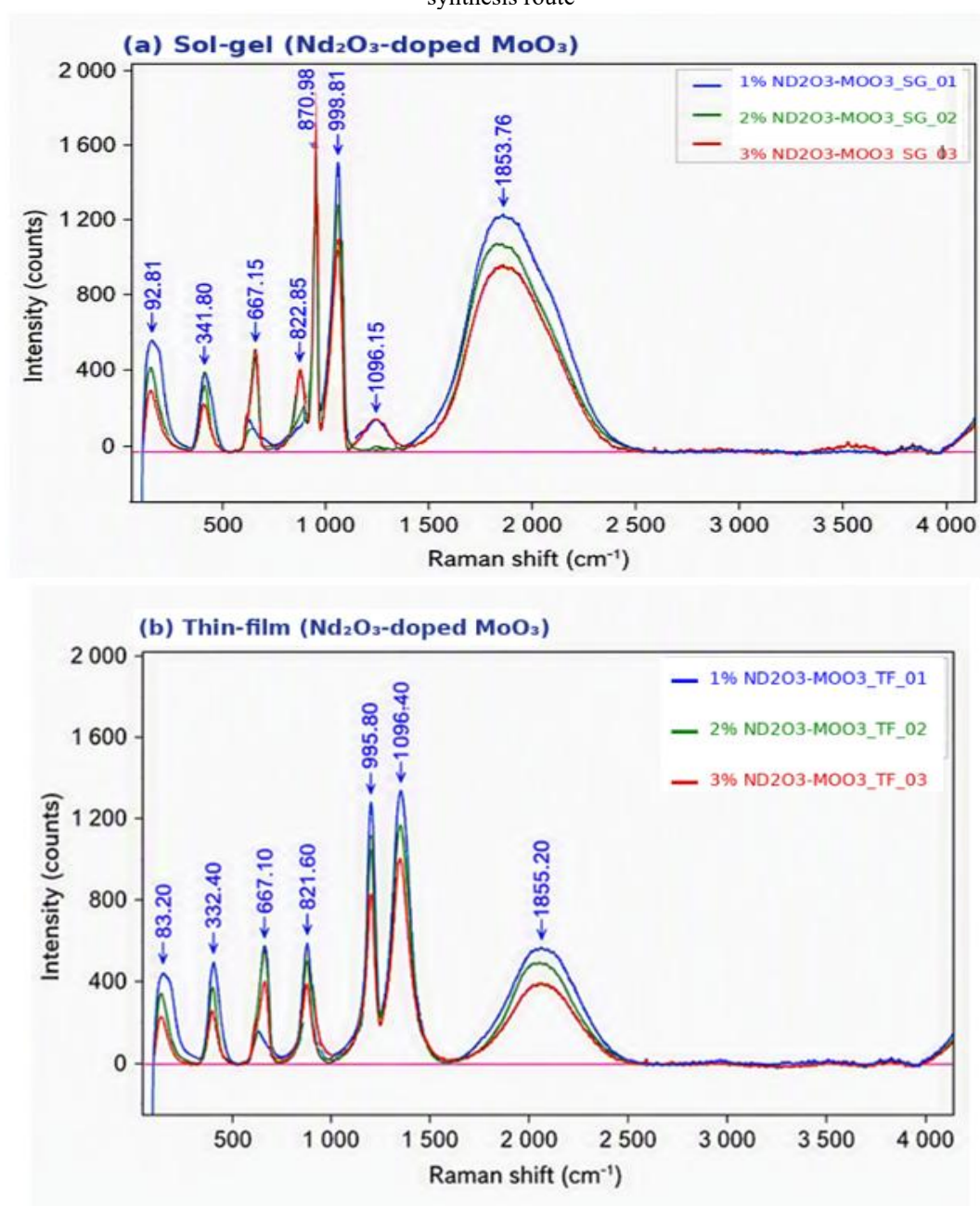


Figure 7: Raman Analysis (a) Sol-gel Nd_2O_3 -doped MoO_3 Raman spectra for 1%, 2%, and 3% and (b) Thin-film Nd_2O_3 -doped MoO_3 Raman spectra for 1%, 2%, and 3%

Antioxidant Activity:

The in vitro DPPH radical scavenging potential of the nanostructures prepared by both the sol-gel and thin-film routes at 1%, 2%, and 3% Nd concentration is shown in the figure above and the corresponding IC₅₀ values are also summarized in the bar chart (Figure. 8).

In synthesis both, the % inhibition rose with concentration of the samples (200 - 1000 µg/mL) indicating a classical dose-dependent free-radical scavenging response. The scavenging activity increased monotonously with increasing Nd³⁺ doping level (1% doping < 2% doping < 3% doping) for all concentrations of MoO₃ tested for both the routes, suggesting that an increase in the Nd³⁺ doping level increases the radical-scavenging ability of the MoO₃ matrix. Increasing substitution of Nd³⁺ at Mo⁶⁺ sites increases the parity of oxygen vacancies (Nd₂O₃ → 2Nd'_{Mo} + 3O₂ + V_O••), a surface that is known to facilitate redox cycling and scavenging of ROS in oxide nanozymes, much like the well-established Ce³⁺/Ce⁴⁺ mechanism for ceria-based nanozymes.

These results can be quantified directly by the IC₅₀ values (Table 2). Similarly, for the sol-gel route the IC₅₀ values varied from 634 to 479 to 379 µg/mL with increasing amount of doping for Nd³⁺, whereas for the thin-film route, they went from 559 to 479 to 379 µg/mL in the same order of doping concentration — in both cases, confirming that antioxidant potency (inversely related to IC₅₀) increases progressively with the amount of doping for Nd³⁺. Remarkably, their thin-film counterparts exhibit lower IC₅₀ (higher activity) at all doping levels than their sol-gel counterparts (e.g., 3%: 379 vs. 423 µg/mL). This difference was found to be route dependent and consistent with the more amorphous/defect rich nature of the thin films suggested by the XRD and Raman results, and thus more accessible surface area and higher density of under-coordinated, vacancy rich surface sites would be expected to provide a higher number of accessible redox active centers for interaction with DPPH radical in solution phase. All the Nd₂O₃–MoO₃ samples are found to be weaker than the ascorbic acid reference (IC₅₀ = 285 µg/mL) at all levels of doping, and the fact that the activity of the samples increases regularly with increasing Nd³⁺ content and further increases from sol-gel to thin-film samples provides a clear empirical relationship between structure, defect content, and activity throughout the data set.

Sr. No.	Sample	Concentration (µg/ml)	1%	2%	3%	% inhibition	IC ₅₀ (µg/mL)
1	Control	-	-	-	-	-	-
2	Standard Ascorbic Acid	200	-	-	-	42.5 ± 1.2	285
		400	-	-	-	58.7 ± 1.1	
		600	-	-	-	72.4 ± 1.0	
		800	-	-	-	84.3 ± 0.9	
		1000	-	-	-	93.8 ± 0.8	
3	Sol-gel route Nd ₂ O ₃ -doped MoO ₃	200	24.8	29.6	34.2	-	634
		400	36.5	42.8	48.6		
		600	48.3	55.7	62.5		
		800	59.6	67.8	74.9		
		1000	68.5	76.4	84.7		
3	Thin-film route Nd ₂ O ₃ -doped MoO ₃	200	27.5	32.4	37.8	-	559
		400	39.8	46.4	53.2		
		600	52.1	60.8	67.4		
		800	64.5	72.8	80.6		
		1000	73.6	81.9	89.3		

Table 2: Antioxidant Activity of Nd₂O₃-Doped MoO₃ Nanoparticles by DPPH Assay

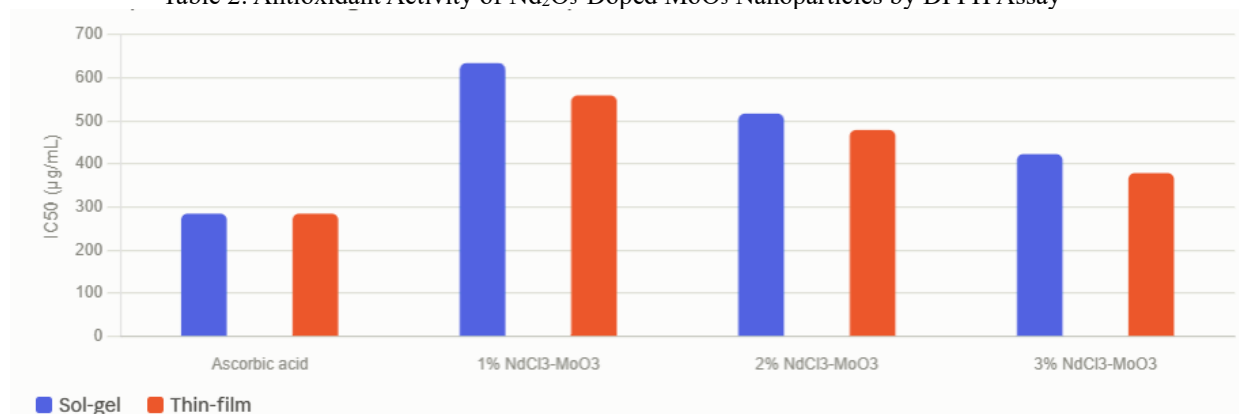


Figure 8: IC₅₀ Comparison (lower = stronger antioxidant activity) of Nd₂O₃-doped MoO₃ nanostructures

Photosensing Activity:

The photoresponse of the samples prepared both routes using Nd_2O_3 – MoO_3 ratio were evaluated by recording dark current (I_{dark}) and photocurrent (I_{ph}) by giving illumination to the samples and using the equation $\% \text{Photoresponse} = [(I_{\text{ph}} - I_{\text{dark}})/I_{\text{dark}}] \times 100$ (Figure. 8).

For the sol-gel route, the dark current increases only marginally with doping ($1.25 \rightarrow 1.31 \rightarrow 1.39 \mu\text{A}$), while the photocurrent rises much more steeply ($4.85 \rightarrow 6.74 \rightarrow 8.46 \mu\text{A}$), driving the photoresponse from 288% at 1% to 415% at 2% and 509% at 3% Nd. The dark current showed the same qualitative trend ($1.46 \rightarrow 1.54 \rightarrow 1.61 \mu\text{A}$) for a systematic increase to higher absolute values, the photocurrent showed a comparable qualitative trend ($6.91 \rightarrow 9.28 \rightarrow 11.54 \mu\text{A}$), but with higher absolute values, and the photoresponse increased from 373% to 503% to 617% along the same sequence of doping values.

There are two consistent trends. First, the increase in dark current is only small as the Nd^{3+} content is increased in both routes, whereas the increase in photocurrent is quite large that is, the increase in the photoresponse is primarily due to the increase in the number of photogenerated carriers that are collected under illumination, not to an overall increase in background conductivity. This is like what was found for the oxygen-vacancy population created by Nd^{3+} substitution as an intermediate/transition trap/recombination-efficient state and only a small parasitic component to the dark leakage current. However, despite the higher-amorphous-phase content in the XRD pattern of the thin films and the higher-defect-density surface, revealed by Raman/XPS, the thin-film samples outperform the sol-gel powders at all doping levels e.g. at 3%: 617% vs. 509%). This apparent contrast is actually consistent with the geometry of the film itself: a thin and supported by a substrate film offers a much shorter path along which the carriers can travel to the collecting electrodes, and a much larger surface to volume ratio for photon absorption and defect-assisted carrier generation (and hence the net photoresponse) than a compacted bulk powder pellet. This same area dependence on surface is employed to elucidate the enhanced anti-oxidative capability of the thin film, and is again followed, such that the same theme of trade-offs of surface sensitivity for both the photogenerated-carrier-based and radical-scavenging-based functional responses is maintained.

Overall, the photosensing results confirm that the amount of photosensing photoresponse monotonically increases with Nd^{3+} doping level for both routes, in accordance with the defect-assisted photocarrier generation, and TF-3 (3% Nd, thin film) is a superior photosensing platform than the sol-gel powder when compared to all doping levels explored, with a photoresponse of 617%.

Sample	Concentration ($\mu\text{g/ml}$)	Dar Current (μA)	Photocurrent (μA)	Photoresponse (%)
Sol-gel route Nd_2O_3 -doped MoO_3	1%	4.85	4.85	288
	2%	6.74	6.74	415
	3%	8.46	8.46	509
Thin-film route Nd_2O_3 -doped MoO_3	1%	1.46	6.91	373
	2%	1.54	9.28	503
	3%	1.61	11.54	615

Table 3: Photosensing activity of Sol-gel and thin-film route of Nd_2O_3 -doped MoO_3 nanostructures

Conclusion:

From a common precursor chemistry, using ammonium heptamolybdate and $\text{NdCl}_3 \cdot 6\text{H}_2\text{O}$, successful synthesis of nanostructures of MoO_3 doped with Nd^{3+} at 1% 2% and 3% was carried out by two separate routes, namely by sol-gel process powders and by spin coating process thin layer, so that the influence of the synthesis route could be separated from the influence of the dopant concentration. XRD of 2% compositions confirmed that the samples were phase-pure and that there was no detectable secondary dopant phase formed, with a highly crystalline orthorhombic α - MoO_3 for the sol-gel powder (MN-2) compared to a less crystalline, hexagonal-leaning structure with a higher amorphous background for the thin film (TF-2), which was attributed to the different calcination/tempering processes experienced by the bulk furnace sample compared to the substrate-confined thin film sample. XPS confirmed that Mo was predominantly Mo^{6+} and Nd was substituted in both routes, as Nd^{3+} , with a resolvable O 1s peak associated with oxygen vacancies providing direct evidence of the Nd^{3+} -substitution/oxygen-vacancy defect-compensation mechanism proposed to govern this system. Incorporating these results, EDS confirmed no detectable chlorine residual or consistent dose-dependent increase in Nd signal as a function of the number of Nd^{3+} atoms in the lattice for both routes, and Raman spectroscopy confirmed a consistent set of observation features which included a displacement of the terminal $\text{Mo}=\text{O}$ stretching mode to higher energy, as well as a growing defect-related spectral envelope with increasing Nd^{3+} content for both routes, independent of XRD and XPS observations of vacancy-mediated lattice distortion. The way in which the powders were made (sol-gel) and the thin films were made (doping) had a significant effect on the morphology of the final products, as shown by the FESEM images, which also indicated a significant morphological evolution from fine nanoparticulate/nanofibrous grains in the sol-gel powders to well-faceted microplatelets that gradually coarsen into more irregular shapes due to the progressive addition of doping for thin film formation.

Functionally, both photosensing and antioxidant performance of the nanostructures of Nd_2O_3 - MoO_3 improved monotonically with increasing amount of Nd^{3+} in both routes, thus correlating the oxygen-vacancy-rich defect

chemistry deduced from the structural and spectroscopic results to measurable device- and radical-scavenging-level results. The photoresponse was found to increase along the 1-3 at. % doping series in both the sol-gel and thin-film cases and was shown to be higher for the latter at each doping level due to a lower carrier transit length and higher surface-to-volume ratio of the thin-film geometry. Similarly, the DPPH radical scavenging IC₅₀ value decreased from 634 to 423 µg/mL (sol-gel) and from 559 to 379 µg/mL (thin film) with the same doping level and level of concentration, with a similar trend being seen with higher activity with the thin film sample at all concentrations reflecting the higher defect density and larger accessible surface area. Not only are both application outcomes found on the same trend, which is clearly dependent on the route of delivery and the used dopant, but both are also induced from completely different physical mechanisms (photogenerated carrier transport versus radical-scavenging redox chemistry), which yields an appropriate degree of internal validation that the oxygen vacancy formation induced by Nd³⁺ is the common structural driver involved in both functional enhancements. The work presented here provides an overall basis for developing Nd₂O₃–MoO₃ as a multifunctional nanomaterial platform which can be tuned structurally, morphologically, and defectively by adjusting the dopant concentration and synthesis route to optimize its efficient performance for both optoelectronic (photosensing) and biochemical (antioxidant) applications. The best samples for both applications were those from the thin-film route (TF-3) with 3% Nd doping for MoO₃, suggesting it to be a promising candidate for further development. It should be extended beyond 3% Nd content to determine if there is an antioxidant/activity/crystallinity trade-off ceiling, and complementary techniques could be used to quantify the band-gap and defect-state density along with cell-based assays to make the antioxidant findings applicable to biomedical applications, while long-term photodetector stability and retention of antioxidant activity under ambient storage conditions should also be evaluated.

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Conflict of Interests:

The authors declare that there is no conflict of interest. The co-author has seen and agree with the contents of the manuscript and there is no financial interest to report. We certify that the submission is original work and is not under review at any other publication.

Author Contributions:

All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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