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Synthesis and Characterization of Nanostructured Nickel Tellurides for Exploring Their EMI Shielding Behavior and Thermoelectric Properties

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ABSTRACT: Amongst the promising transition-metal chalcogenides, nickel tellurides at nanoscale are widely recognized for their distinctive structural and electronic properties and multifunctional behavior that enables their use in a broad range of modern technologies. Nickel tellurides with customized properties can be produced by various techniques that include hydrothermal and solvothermal routes, being the most popular, etc. The present findings are based on direct synthesis of phase-pure nickel telluride, with varying nonstoichiometric compositions. The progress of the experiment was assessed by UV-visible spectroscopy, and the so-synthesized samples were named as DB-4 (NiTe₂), DB-6 (Ni₂Te₃), and DB-7 (NiTe). The formation of different near stoichiometric nickel telluride(s) was finally confirmed by X-ray diffraction measurement. The diameters in the range of about 8–14.1 nm with lattice spacings of about 0.29 nm at the (101) plane was estimated for the so-obtained nanostructured nickel tellurides. X-ray photoelectron spectroscopy (XPS) analysis revealed the elemental composition and chemical states of the nickel telluride. HRTEM and FESEM imaging showed directional clustering possibly due to the magnetic character of the nanoparticles. Magnetic property evaluation showed weak magnetic behavior, indicating it to be ferromagnetic in nature. The black powders were converted to (polyvinyl alcohol) PVA films to realize their low X-band EMI shielding effectiveness (SE) of about 3.1 dB, which showed an increasing trend with percentage filler. Similarly, their thermoelectric data suggest that they are materials that can be tailored for potential applications.

KEYWORDS: Transition metal chalcogenides; Nickel tellurides; synthesis; EMI shielding; thermoelectrics

1 Introduction

Over the past several decades, special attention has been focused on the research that has propelled the deeper understanding and utilization of transition metal chalcogenides (TMCs) as versatile functional materials for diverse technological applications [1–4]. Amongst the transition metal tellurides, nickel telluride(s) have technologically become very attractive due to its phase diversity and physical properties. Historically, metal tellurides were investigated in the context of energy materials, e.g., thermoelectric and superconducting materials due to their unique electronic structures [5]. Nickel tellurides are viewed as emerging transition-metal chalcogenides with tunable electronic and magnetic behavior, making them valuable functional materials. These compounds show fast electron-transfer ability and adjustable band structures to suit the requirements and utility in electrocatalysis and sensing. Overall, nickel tellurides are increasingly recognized as versatile, stable, and high-performance materials with strong potential for next-generation nano-engineered technologies [6–10], viz., electrochemical processes, including supercapacitors, batteries, and hybrid energy storage systems [11–13], such as asymmetric supercapacitors, urea

oxidation, and hydrogen evolution reactions (HER) [14–17]. It has been opined that the electronic interaction between nickel and tellurium may enhance charge transport properties to make the material highly suitable for catalytic reactions. Three different phases of nonstoichiometric nickel tellurides have often been described, e.g., NiTe, NiTe₂, and Ni₃Te₂. The polymorphs of two important crystal structures of NiTe₂ (cubic) and Ni₃Te₂ (hexagonal) are well discussed [14]. At the nanoscale, nickel tellurides reveal fascinating traits and make Ni-Te nanostructures highly suitable for thermoelectric devices also. Researchers have exclusively studied NiTe rods that have been synthesized via a hydrothermal method involving Ni(OH)₂ and tellurium ions to obtain porous hollow nickel telluride nanosheets with high conductivity and large surface area [14,16]. For electrochemical studies, Manikandan et al. [17,18] reported nickel cobalt telluride nanorods for supercapacitor applications, while various crystal phases such as NiTe and Ni_{2.86}Te₂ in the form of nanosheets were synthesized and exploited for HER by Xiang et al. [19]. Hexagonal NiTe and trigonal NiTe₂ nanorods [20] have been reported via effective reduction of metal salts to demonstrate semiconducting behavior. Liu et al. [21] reported an *in situ* synthesized NiTe₂ nanocomposite with nickel selenide on nickel foam that exhibited superior supercapacitor performance as electrodes. Alteration in synthesis method was explored by Zhang et al. [22] by using microwave energy to isolate NiTe nanosheets on nickel foam in one step for supercapacitor applications. Zhang et al. [23] reported hydrothermally synthesized hollow NiTe₂ nanotubes as bi-functional materials for both hydrogen evolution reaction (HER) and supercapacitor applications. Despite immense studies on nickel tellurides, no literature exists on their exploitation as electromagnetic interference (EMI) shielding materials.

Of late, researchers have demonstrated the utility of metal chalcogenides for EMI, where several types of chalcogenides, especially metal selenides and tellurides, are studied. EMI shielding of 50.1–55.5 dB in the X-band was reported by Chen et al. [24] for antimony telluride (Sb₂Te₃) nanosheets with a thickness of 1.36 mm. Al-Asbahi et al. [25] enhanced EMI shielding performance of about 60.88 dB at 12 GHz by chemically treating flexible Te/PEDOT: PSS film, relying on its adsorption property that makes it highly conductive and a good candidate for wearable electronics. In our recent reports, we have described the use of silver-doped binary and ternary metal chalcogenides as an efficient active EMI shielding material with reasonable shielding performance [26–29]. According to Kumar et al. [30], Ag@PANI/TeO₂ ternary hybrid nanocomposites showed EMI SE of roughly in the –5 to –9 dB range in the S-band (2–3 GHz frequency). As EMI studies for metal tellurides, especially of nickel tellurides, are not documented in the literature, the same is investigated to enrich the field of metal tellurides and widen their versatility. The EMI shielding effectiveness may be directly proportional to the size and morphology, as the film formation will greatly depend on the nature of nanomaterials.

Environmental safety and performance of electronic components may be adversely impacted due to EM waves amounting to radiation, conduction, and induction. EMI shielding effectiveness (SE) is mainly due to three mechanisms; however, magnetic properties may also play a role [31–34]. The total SE can be derived from reflection and absorption losses, and multiple internal reflections become important. Reflection loss increases with higher conductivity and lower permeability. Further understanding about the reflection losses arises from the fact that for electric fields (E-fields), reflection increases at lower frequencies and shorter distances, and for magnetic fields (H-fields), reflection decreases at lower frequencies. However, for plane waves, reflection increases as frequency decreases. Similarly, basic understanding of absorption losses is necessary for interpreting material's behaviour; thus, attenuation of the wave induces current causing ohmic and heat losses. This attenuation is known as absorption loss, which is proportional to $A \propto \sqrt{\mu\sigma}$. Therefore, good absorbers should have high conductivity, magnetic permeability, and sufficient thickness. Overall, the absorption loss increases with increasing thickness, permeability, and conductivity.

Materials with high permeability, such as iron or ferrites, are especially effective for magnetic field shielding. Multiple reflections may occur within the shield due to repeated reflections at internal surfaces. Their contribution depends on the magnitude of absorption loss. The higher absorption loss leads to negligible internal reflections. Overall, effective EMI shielding can be achieved through dielectric field absorption (using conductive materials) or magnetic field absorption (using high-permeability materials). Therefore, an ideal shielding material generally combines high conductivity, high permeability, and adequate thickness.

Similarly, there are other potential applications of metal tellurides, e.g., thermoelectric devices. Thermoelectric applications of metal tellurides are part of modern technological advancement where thermal energy is converted to electrical energy through the Seebeck effect. Such technologies use energy from body movement or heat for conversion to electricity through devices like thermoelectric generators. When a temperature gradient is created across a thermoelectric material, an electric potential is created due to movement of charge carriers to the cold region from the hot region. Thermoelectric generators are widely studied for waste heat recovery applications [35]. Thermoelectric devices include thermoelectric refrigerators and generators, which are applicable in solid-state cooling/heating and power generation, respectively [36]. The suitable thermoelectric materials are evaluated based on the figure of merit zT , a dimensionless entity, and are expressed by the following equation:

$$zT = \frac{S^2 \sigma T}{\kappa}$$

Materials with a high Seebeck coefficient (S), high electrical conductivity (σ), low total thermal conductivity (κ) and temperature (T) are well suited for thermoelectric applications. The higher is the electrical conductivity, higher will be zT . The term $S^2 \sigma$ in the equation is collectively known as the power factor. In order to achieve the right materials, selection of correct the material synthesis is important.

2 Experimental Section

2.1 Materials and Methods

Nickel chloride (Sigma-Aldrich, 98%), hydrazine hydrate (80% Qualigens), and sodium tellurite (Sigma-Aldrich) were acquired from respective suppliers. Deionized water was obtained from local sources. The experiments were conducted under an inert atmosphere of nitrogen gas. X-ray diffraction patterns were obtained using Cu-K α ($\lambda = 1.541 \text{ \AA}$) on a Proto Benchtop XRD (Canada). XPS (Omicron ESCA Oxford Instrument Germany) and HRTEM (JEOL JEM 2100 PLUS) analyses were carried out by Sprint Technology, Mumbai, India. FESEM (Carl Zeiss SIGMA machines) images were obtained from machines housed at DIAT. UV-visible spectra were recorded in water between 300 and 800 nm on an Analytik Jena spectrophotometer. FTIR spectra were recorded in the mid-IR range from 400 and 4000 cm^{-1} on the PerkinElmer Spectrum two machine. X-band EMI shielding (8.2–12.4 GHz) was evaluated using VNA model no. N5222A of Keysight Technologies. HP4294A (VSM) operating at a frequency of 10 KHz, was used for the magnetic measurement. Thermal conductivity measurement was done on the Thermtest MP-V instrument (Canada), and for thermoelectric studies, dried powder samples were converted to PVA films and were cut into desired dimensions. The thermoelectric studies were performed at room temperature (25°C) by one of the authors at HEMRL, Pune.

2.2 Synthesis of Nickel Telluride

Nickel telluride (NiTe , NiTe_2 , and Ni_2Te_3) nanoparticles were synthesized via a wet-chemical route using aqueous precursors. In a clean round-bottom flask, stoichiometric amounts of NiCl_2 and Na_2TeO_3

(0.345 g & 0.59 g for NiTe, 0.4128 g & 1.4 g for NiTe₂, and 0.518 g & 1.329 g for Ni₂Te₃) were dissolved in deionized water (about 50–60 mL) with rapid stirring at room temperature for the dissolution of metal ions and tellurium species. The reaction mixture was gradually heated to promote ion mobility and facilitate subsequent reduction. After approximately one hour, 10–12 mL of hydrazine hydrate (80%) was introduced dropwise to simultaneously convert Te *in situ* to Te²⁻ as well as reduce nickel salt. The reduction and nucleation processes were completed in 2–3 h at about 100–110°C. The experiment was monitored by UV-visible spectroscopy to check if the absorption band of nickel chloride disappears for that will confirm complete conversion of the precursor to Ni-Te(s). Following completion and cooling of the reaction mixture, it was centrifuged twice with DI water and ethanol to collect black powder, which was dried in an electric oven. The samples were named as DB-4 (NiTe₂), DB-6 (Ni₂Te₃) and DB-7 (NiTe).

2.3 Synthesis of Nickel Nanoparticles

NiCl₂ (4.0 g) was taken in a two-neck round-bottom flask with deionized water (75 mL) and kept on a magnetic stirrer for vigorous stirring. After 10 min, heating was switched on for localization of ions. In order to dissociate Ni⁺ and Cl⁻, an appropriate reducing agent, such as hydrazine hydrate (80%, 15–18 mL), was added drop-wise after 30 min. The reducing agent, 80% hydrazine hydrate, was deliberately added in excess for further formation of Ni-nanoparticles. The nickel nanoparticles formed a mirror inside the round-bottom flask due to their metallic nature, and the formation of metallic nickel nanoparticles was confirmed when the particles started getting attracted to the magnetic needle. The precipitate was centrifuged and followed by washings at twice with ethanol & water. The product was dried in an electrically heated oven.

2.4 Synthesis of Nickel-Rich Nickel Tellurides

Nickel-rich nickel telluride was prepared using the hydrothermal method as per the protocol followed in Section 2.2. Thus, there prepared, practicing *in-situ* and *ex-situ* approach. For *in-situ* synthesis, stoichiometric amounts of NiCl₂ and Te-source (1.626 g & 1.412 g; 4:2 ratio, i.e., 2:1) and 2.032 g & 1.412 g for 5:2 ratio) were introduced to deionized water (50–60 mL) to obtain black powders. Alternatively, pre-synthesized nickel telluride powder (0.5 g) and nickel nanoparticles (0.5 g; 1:1 w/w) were homogenized via hand grinding in a mortar and pestle to prepare a nickel-rich sample for further studies.

2.5 Synthesis of Silver-Containing Nickel Tellurides

In a round-bottom flask, NiCl₂ and a Te source (0.41 g & 1.4 g) were reacted in deionized water and kept for vigorous stirring. Uniform heating was introduced to the reaction after 10 min of stirring. 80% hydrazine hydrate (reducing agent, 10–12 mL) was added drop-wise in the RB for reduction of the ions after 30 min. Before completion of the reduction and formation of final compounds, silver nitrate (0.1 g) was introduced after 1 h and heated for 3 h, and the flask was left to cool. The so-formed precipitate was subjected to centrifugation using ethanol and water, and the powder was dried in an electrical oven.

2.6 Preparation of Nickel Telluride-Polyvinyl Alcohol (PVA) Film

PVA (2.0 gm) was suspended in 100 mL of deionized water and was irradiated with microwave energy for 5–10 min to obtain a transparent viscous liquid. This was followed by the addition of 0.2 gm of nickel telluride (NiTe₂ and Ni₂Te₃) and the mixture was sonicated and stirred for half an hr. The homogeneous dense liquid was poured into a petri dish and was left for 48 h to evaporate water, by which time the dense liquid was converted to a film of the diameter of 2" with an approximate thickness of about 1 mm. The

so-generated films, i.e., NiTe₂/PVA and Ni₂Te₃/PVA film, were further dried at about 50 degrees for 1–2 h before using them for thermoelectric studies.

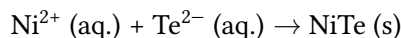
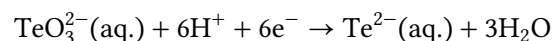
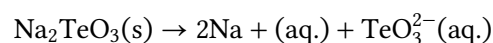
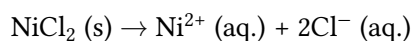
2.7 Preparation of Films for Thermoelectric Studies

PVA (1.0 gm) was suspended in 100 mL of deionized water and was irradiated with microwave energy for 5–10 min to obtain a transparent viscous liquid. This was followed by the addition of 0.2 gm of nickel telluride (NiTe₂ or Ni₂Te₃), and the mixture was sonicated and stirred for half an hour. The homogeneous dense liquid was poured into a petri dish and was left for 48 h to evaporate water and the dense liquid was converted to a film with an approximate thickness of about 0.05 cm. The so-generated NiTe₂/PVA and Ni₂Te₃/PVA films were carefully cut using a cutter into a rectangular size of 1.0 cm X 0.3 cm X 0.05 cm. The samples were analyzed for thermoelectric studies.

3 Results and Discussion

3.1 Synthesis and Characterization

The reaction of nickel metal salt (NiCl₂) was carried out in an aq. medium under a reducing environment using hydrazine hydrate via a modified method of hydrothermal reaction, which normally takes 12 h [20]. In the present case, the nickel precursor was reacted with sodium tellurite. The overall reaction occurs in two stages wherein in the first stage the respective ions are formed via a bond dissociation, and in the second stage they are reduced by the reducing agent. Eventually the reaction mixture was gradually heated to promote ion mobility and facilitate subsequent reduction. After approximately one hour, a suitable reducing agent, hydrazine hydrate, was introduced dropwise to convert Te to Te²⁻. Finally, the divalent Ni and Te ions react with one another to yield a black precipitate of nickel telluride that was dried and analyzed. A similar approach has been practiced by other researchers too [4]. However, in the present work, the precursors and the steps were designed in such a way as to obtain the nanopowder in a single step and within 3–4 h. Overall, reactions are presented below



The tellurite ion formation depends upon the concentration and environment of the reducing agent. Experiments even without a nitrogen atmosphere were good enough to produce the nanostructured nickel tellurides. The current method eliminated various complex reaction assemblies and high-pressure chemical reactions. Non-stoichiometric synthesis in order to understand the effect of excess nickel or tellurium was useful from an application point of view; thus, these samples were also prepared following *in situ* and *ex situ* methods. For the *in situ* method we, essentially used an excess of nickel precursor; however, the *ex-situ* sample was prepared by first preparing nickel nanoparticles, followed by their mixing with pre-prepared nickel telluride, and were homogenized via hand grinding to obtain nickel-rich nickel telluride.

The reaction progress was assessed by recording the UV-visible spectrum of final aliquots in water that showed no absorption in the region of nickel chloride. It was observed that the initial absorption

peaks/bands in nickel chloride appeared at 395 nm with broad patterns at about 650 and 725 nm (Fig. 1). As the synthesis progressed, these peaks/bands steadily weakened and eventually disappeared, confirming the completion reaction. The complete fading of the absorption bands indicated that the precursor was fully consumed during the transformation of Ni^{2+} ions to nickel telluride(s) [37].

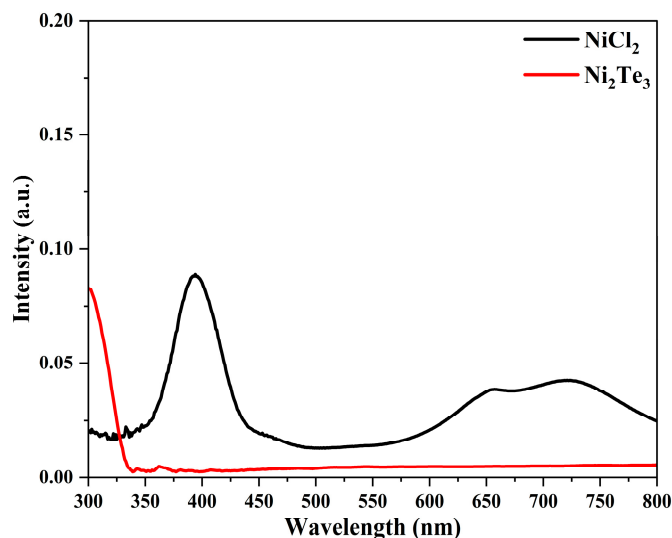


Figure 1: Typical UV-visible spectra of NiCl_2 and Ni_2Te_3 .

The powder XRD measurement was performed for understanding the phase formation, structure, and crystallite size of the synthesized nickel telluride(s) samples. Indeed, the recorded XRD patterns confirmed the successful formation of different stoichiometric nickel telluride(s) with good crystallinity and were in close agreement with the standard literature data corresponding to NiTe , NiTe_2 and Ni_2Te_3 and their JCPDS file No. 00-038-1393, JCPDS file No. 00-008-0004 and JCPDS file No. 00-002-0810, respectively [37,38]. The crystallite size was estimated using the Scherrer equation considering the half width of the prominent diffraction peaks, and the calculated values are presented in Table 1 (Fig. 2). For sample DB-4 (NiTe_2), distinct diffraction peaks were located at 2θ values of 31.64° , 43.62° , 47.12° , and 59.16° , corresponding to the crystallographic planes (101), (102), (110), and (112), respectively [20]. The corresponding interplanar spacing (d-spacing) values were calculated as 0.28, 0.21, 0.19 and 0.16 nm which were matched with the literature values. The crystallite sizes calculated for these reflections were found to be 17.1 nm, 9.4 nm, 19.2 nm, and 10.8 nm, respectively, with an average size of 14.1 nm. The sharp peaks are correlated with the crystalline nature of nickel telluride(s). Similarly, the XRD pattern of sample DB-6 (Ni_2Te_3) exhibited diffraction peaks at 2θ values of 31.31° , 43.01° , 46.70° , and 58.37° , corresponding to the crystallographic planes (101), (102), (110), and (201). The calculated d-spacing values were 0.29, 0.21, 0.19 and 0.16 nm, respectively. The crystallite sizes estimated from these peaks were 11.8 nm, 7.0 nm, 9.9 nm, and 4.1 nm, resulting in an average size of 8.2 nm. In the case of sample DB-7 (NiTe), the diffraction peaks appeared at 2θ values of 30.97° , 42.77° , 46.09° , and 58.02° , which correspond to the crystal planes (101), (102), (110), and (201), respectively [15]. The associated d-spacing values were determined to be 0.29, 0.21, 0.20 and 0.16 nm. The crystallite sizes calculated for these reflections were 12.2 nm, 7.8 nm, 10.7 nm, and 3.9 nm, respectively, with an average crystallite size of 8.65 nm. The variation in peak intensity and crystallite size among the samples indicates the influence of composition and synthesis conditions on the structural properties of nickel telluride nanostructures. Thereby validating the phase formation and structural integrity of the synthesized materials. Non-stoichiometric samples were similarly analyzed by XRD, and each one of them

was compared with the originally synthesized nickel telluride sample DB-4 (NiTe_2). The XRD pattern of so-generated nickel-rich NiTe_2 showed all relevant peaks for NiTe_2 as well as some overlapped diffraction peaks for nickel nanoparticles, as both materials have reflections at similar 2θ values. Fig. 3 shows the XRD pattern for such samples. It is observed that hand-ground-prepared nickel-rich NiTe_2 essentially showed the same reflections.

Similarly, other experiments were conducted that are essential to drive conductivity in nickel telluride, e.g., the addition of silver was carried out. The XRD of such a sample (Fig. 4) confirmed the presence of an Ag-containing phase rather than definitive substitution or incorporation of Ag into the NiTe_2 lattice and showed the signature of the presence of silver. It is indeed found that the synthesis of NiTe_2 is reproducible even when a silver precursor is present for the synthesis of *in-situ* doped NiTe_2 . This, therefore, establishes the authenticity of the synthesis method, as the findings are well compared with NiTe_2 (DB-4). As expected, the XRD pattern revealed a characteristic silver peak at 2θ values of about 38.9° for the FCC crystal structure. Other peaks for silver were not so prominent; however, all peaks due to nickel telluride chosen for the preparation of hybrid material via silver doping were present and mostly overlapped.

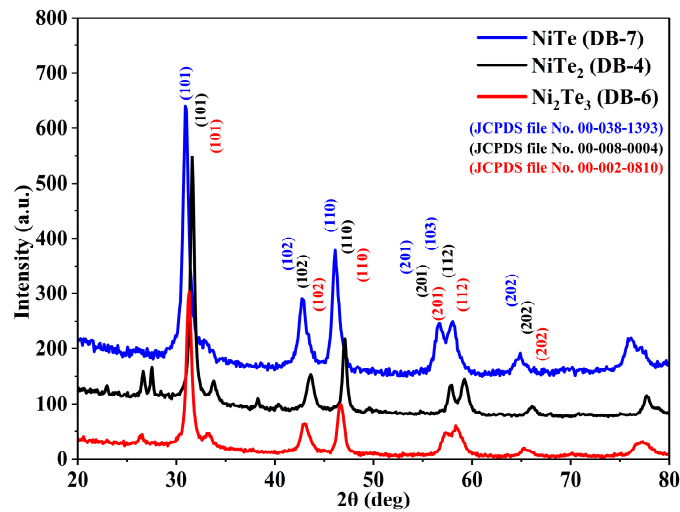


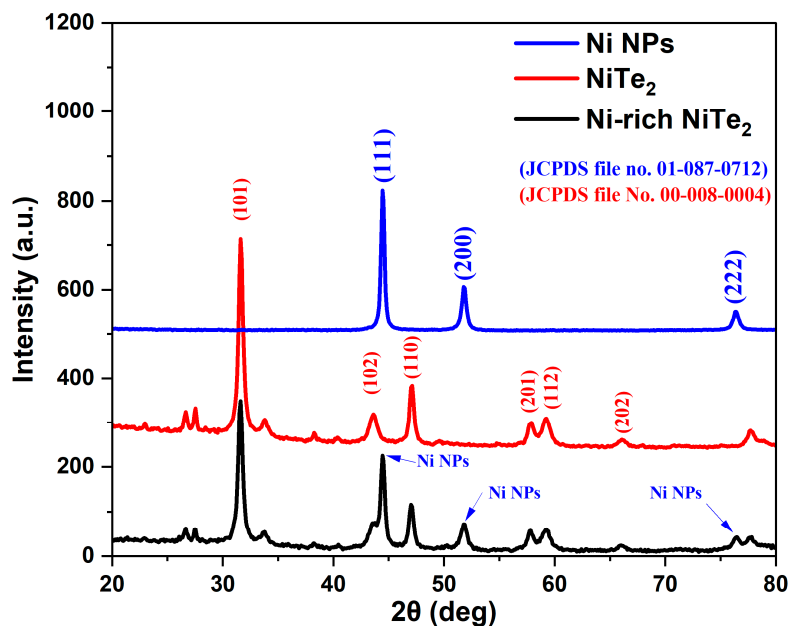
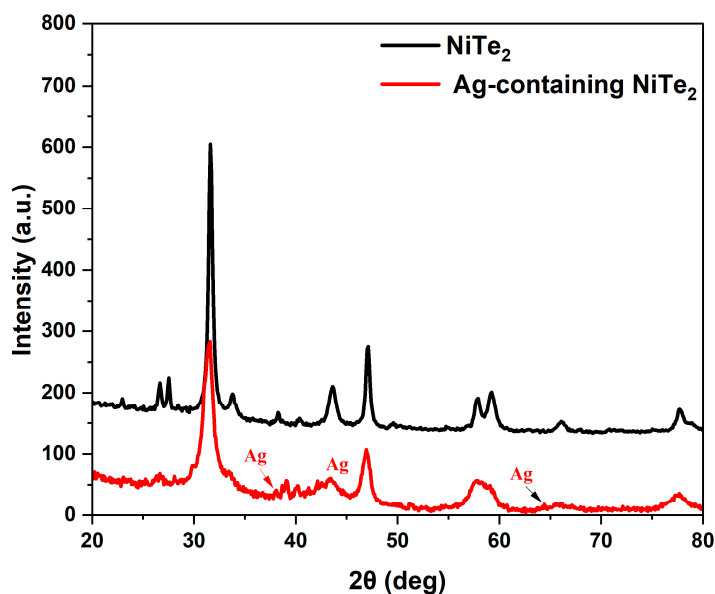
Figure 2: XRD pattern of nickel telluride of three types (a) NiTe (b) NiTe_2 (c) Ni_2Te_3 .

Table 1: XRD data and calculation of crystallite size and lattice-spacings.

S. No.	2θ (deg)	FWHM	d-Spacing (nm)	Crystal Planes	Crystallite Size (D) in nm
DB-4 (NiTe_2)					
1.	31.64	0.4833	0.28	(101)	17.1
2.	43.62	0.9027	0.21	(102)	9.40
3.	47.12	0.4513	0.19	(110)	19.2
4.	59.16	0.8463	0.16	(112)	10.8
Average crystallite size (D)					14.1
DB-6 (Ni_2Te_3)					
1.	31.31	0.6968	0.29	(101)	11.8
2.	43.01	1.2194	0.21	(102)	7.00
3.	46.70	0.8710	0.19	(110)	9.90
4.	58.37	2.1707	0.16	(201)	4.10
Average crystallite size (D)					8.24

Table 1: Cont.

S. No.	2 θ (deg)	FWHM	d-Spacing (nm)	Crystal Planes	Crystallite Size (D) in nm
Sample code			DB-7 (NiTe)		
1.	30.97	0.6742	0.29	(101)	12.2
2.	42.77	1.0909	0.21	(102)	7.80
3.	46.09	0.8094	0.2	(110)	10.7
4.	58.02	2.3281	0.16	(201)	3.90
Average crystallite size (D)					8.65

Figure 3: X-ray diffraction of nickel nanoparticles, nickel telluride (NiTe_2) and Ni-rich NiTe_2 (hand grind).Figure 4: Powder X-ray diffraction analysis of silver doped nickel telluride confirming formation of Ag- NiTe_2 .

Elemental composition and oxidation states of the respective elements in NiTe nanoparticles were investigated by X-ray photoelectron spectroscopy (XPS). The XPS spectra and survey spectrum shown in Fig. 5a–c confirms the presence of Ni, Te, O, and C on the surface of the NiTe (DB-7) NPs. The presence of O and C can be attributed to surface oxidation and adventitious carbon, respectively. The high-resolution Ni 2p spectrum shown in Fig. 5a, reveals the characteristic Ni^{2+} , Ni^0 , and satellite peaks. The peaks at binding energy values of 854.87 eV and 871.9 eV relate to Ni^{2+} 2p_{3/2} and 2p_{1/2}, respectively, and is attributable to the Ni-Te bonding environment in NiTe. The additional peaks observed at 860.86 eV and 879.80 eV can be assigned to the shake-up satellite peaks that normally appear at slightly higher binding energies.

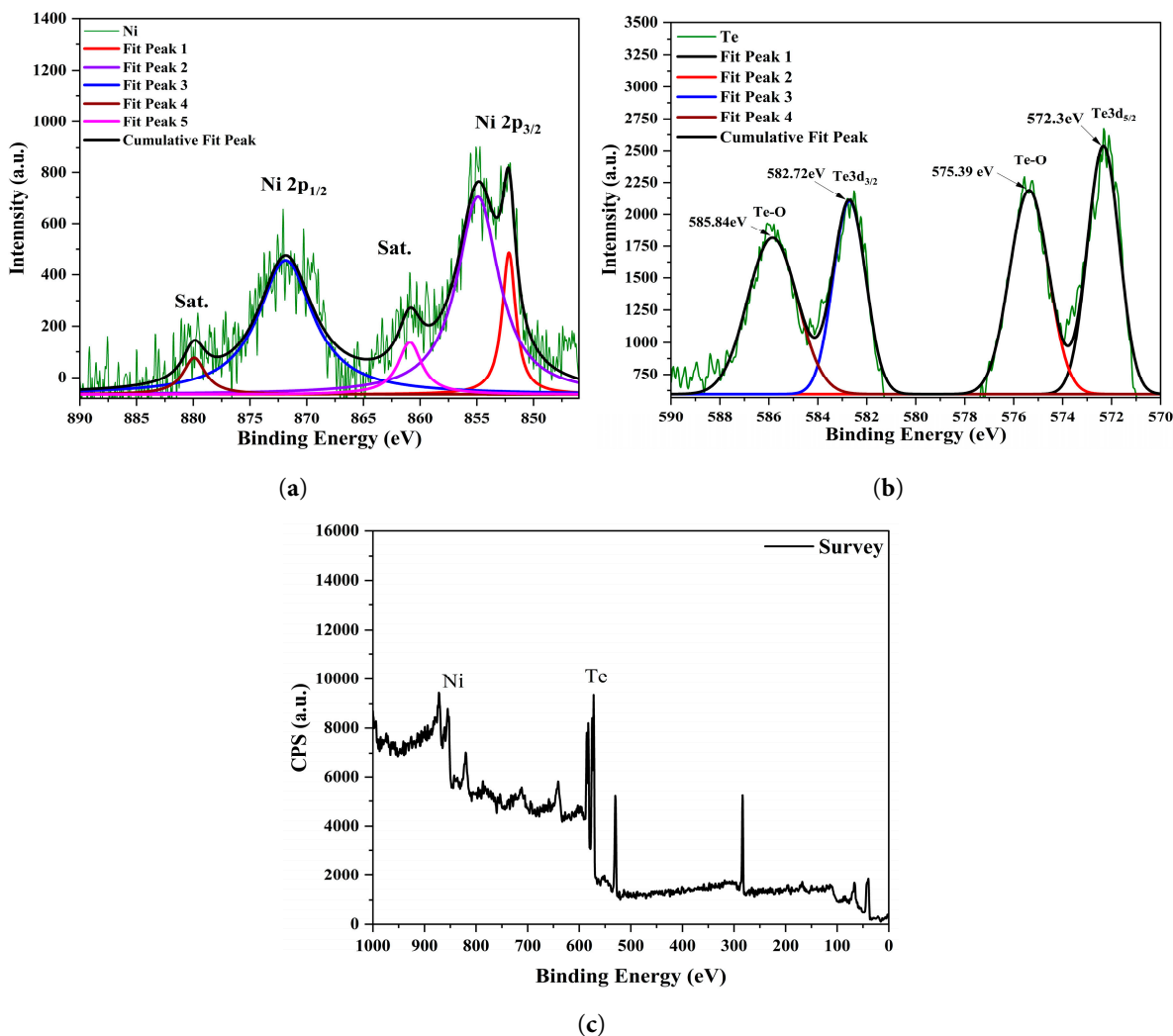


Figure 5: XPS spectra of (a–c) nanostructured nickel telluride (NiTe).

Furthermore, an additional peak at 852.17 eV is attributed to metallic Ni^0 , which may arise due to the formation of nickel nanoparticles during the synthesis that involves hydrazine-assisted reduction of Ni^{2+} ions. Fig. 5b exhibits Te 3d spectrum, revealing the characteristic Te^{2-} and Te^{4+} states. The Te^{2-} 3d_{5/2} and Te^{2-} 3d_{3/2} peaks are located at binding energies of 572.36 eV and 582.72.0 eV, respectively, and are associated with the Ni-Te bonding [19,20]. In addition, the Te^{4+} 3d_{5/2} and Te^{4+} 3d_{3/2} peaks observed at 575.39 eV and 585.84 eV, respectively, indicate the presence of a minor amount of oxidized tellurium

species on the nanoparticle surface. The Te^{4+} contribution in the Te 3d region can be associated with TeO_3^{2-} -containing surface species, suggesting partial surface oxidation of the NiTe NPs upon exposure to atmospheric conditions [21,22]. TeO_3^{2-} -containing surface species may also arise due to the use of Na_2TeO_3 as a Te precursor that might be present at trace levels as an unreacted entity in the final product.

Overall, the evidence for the formation of NiTe NPs has been established by XPS analysis, based on the characteristic Ni^{2+} and Te^{2-} chemical states associated with Ni-Te bonding. The presence of a Ni^0 component is consistent with the reducing conditions employed during synthesis, whereas the Te^{4+} contribution can be attributed to the partial oxidation of the nanoparticle surface or unreacted Te-precursor. Thus, the XPS analysis further supports the successful formation of nickel telluride (NiTe) NPs.

HRTEM images can show lattice fringes, which are the repeating atomic planes inside a crystal. The d-spacing can be matched with the calculated values from the XRD pattern to relate to crystal structure. In the present study, a typical sample DB-4 was analyzed that showed self-assembled particles on a nano-rod template. The morphology is mostly seen to be spherical, but one directional growth of the particles is also observed. This could be due to the fact that initially an anisotropic particle, upon growth, nucleated on the surface of one of the bigger particles, which then grew in a certain direction because of the magnetic nature associated with such materials. The careful examination showed lattice fringes indicating high crystallinity of the material. The electron diffraction analysis showed various spots indicating rings due to crystal planes that co-relate well with the XRD findings. Overall particle diameter seems to be less than 10 nm, which matched well with the findings from XRD (Fig. 6).

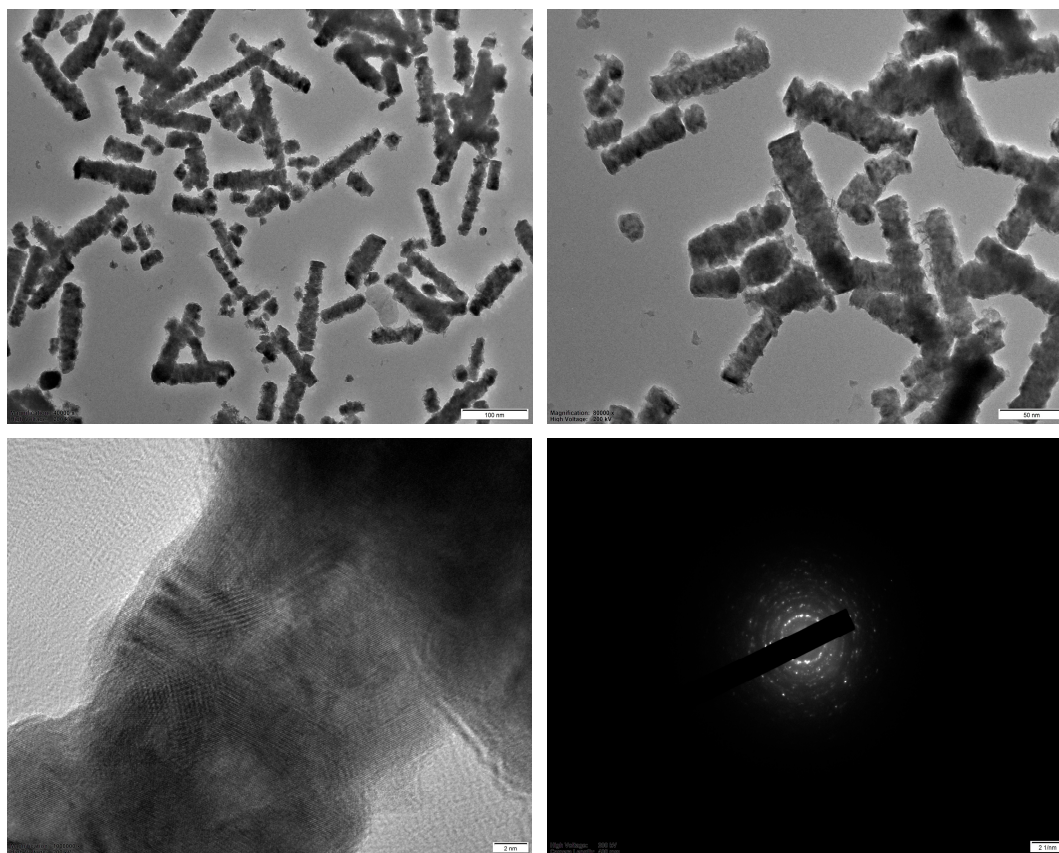


Figure 6: HRTEM images of (NiTe_2) DB-4 sample.

The FESEM images of the so synthesized nickel telluride nanoparticles reveal the formation of highly agglomerated nanostructured particles with mixed morphology. The images recorded at different magnifications indicate that the material consists predominantly of irregularly distributed granular nanoparticles along with rod-like nanostructures. The particles appear densely packed, forming interconnected clusters because of the energetic surfaces of the nanosized material. In the higher magnification images, cauliflower-like agglomerates composed of fine primary nanoparticles can be observed. These secondary structures are formed through the aggregation of smaller crystallites during the hydrothermal growth process. The particle surfaces appear rough and porous, which can provide a larger active surface area beneficial for catalytic and electrochemical applications. The lower image clearly shows the presence of short nanorods or elongated particles distributed throughout the sample. These rod-like structures suggest anisotropic crystal growth, which may arise from preferential growth along certain crystallographic directions during synthesis. The dimensions of the nanorods are within the nanometer range, as indicated by the 200 nm scale bar (Fig. 7). Overall, the FESEM analysis confirms the formation of nickel telluride nanostructures with heterogeneous morphology consisting of nanoparticle aggregates and nanorod-like features. Such morphology is advantageous for applications such as EMI shielding and thermoelectric studies because the interconnected nanostructure can facilitate charge transport and enhance surface-related interactions.

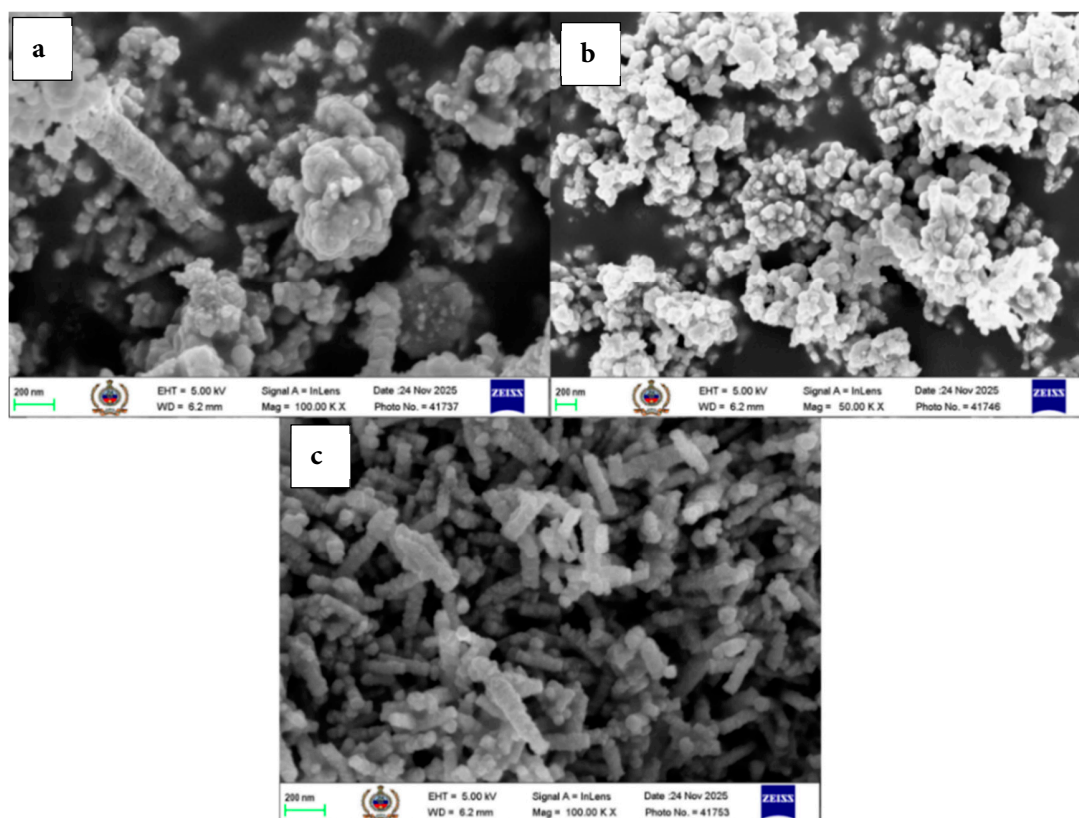


Figure 7: FESEM image of (a) NiTe, (b) NiTe₂ & (c) Ni₂Te₃ sample.

FTIR (Fourier Transform Infrared) spectra showed peaks due to Ni-O and Ni-Te bonds as well as other functionality. The FTIR spectra of pure NiTe₂, nickel-excess nickel telluride (5:3 NiTe₂), and Ag-containing nickel telluride samples (Fig. 8) confirm the formation of nickel telluride-based nanostructures along with functional groups due to environmental moisture adsorption on the surface. Broad absorption bands

attributable to O-H stretching vibrations and are observed in the region of 3200–3600 cm^{-1} , confirming the presence of hydroxyl groups on the surface. Similarly, weak peaks near 1600 cm^{-1} correspond to H-O-H bending of physically adsorbed water molecules. The low-intensity peaks at 445 cm^{-1} (Ag-containing nickel telluride), 604 cm^{-1} (Ni-rich nickel telluride) and 518 cm^{-1} (NiTe) may be associated with Ni-Te vibrational modes, confirming the formation of nickel telluride phases [38]. In comparison with pure NiTe₂, the 5:3 NiTe₂ sample exhibits noticeable variation in peak intensity and transmittance behavior, suggesting changes in stoichiometry and lattice environment due to excess nickel incorporation. For the Ag-containing samples, slight changes in transmittance and slight peak shifts are observed when compared with nickel telluride alone. These modifications indicate successful incorporation of Ag into the nickel telluride, which may influence the local bonding environment. Sample DB Ag-3 shows prominent spectral variation, suggesting stronger interaction of Ag with the NiTe₂.

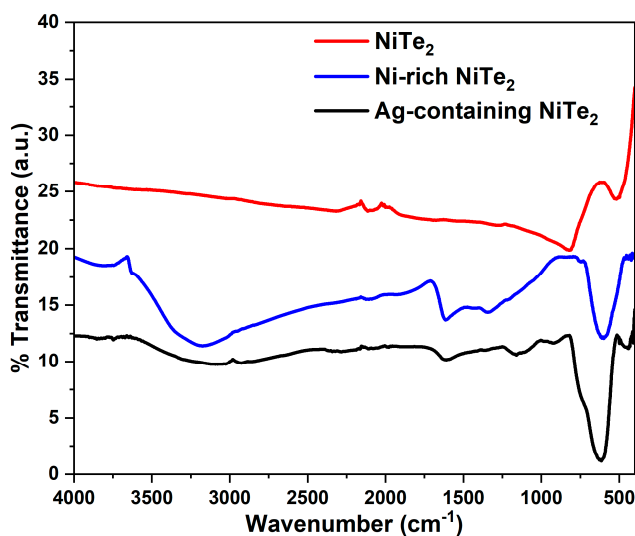


Figure 8: FTIR spectra of (a) NiTe₂, (b) Ni-rich NiTe₂ and (c) Ag-containing NiTe₂.

3.2 Magnetic Property Studies

The magnetic behaviour of a material can be studied by use of Vibrating Sample Magnetometry (VSM), where the sample is in the environment of a magnetic field and is vibrated continuously to measure its magnetic response. The VSM graph mainly shows magnetization (M) versus applied magnetic field (H), called the hysteresis loop. From this graph, important magnetic properties such as saturation magnetization (M_s), remanent magnetization (M_r), and coercivity (H_c) are obtained. Saturation magnetization represents the maximum magnetization achieved by the material. Remanence shows the magnetization left in the sample after removing the magnetic field. Coercivity indicates how much magnetic field is required to make the magnetization zero again. The shape of the hysteresis loop helps determine whether the material is ferromagnetic, paramagnetic, or diamagnetic in nature. Magnetic susceptibility can also be calculated using the simple relation $\chi = M/H$, here, ' M ' is magnetization, and ' H ' is the applied magnetic field.

The researcher reported that not many studies have been performed on magnetic properties of nickel telluride(s) and the available data suggest inconsistency [37,38]. The antiferromagnetic behavior of NiTe has been reported only to be disagreed with by another researcher who reported this material to be paramagnetic [39]. Still further research claimed the material to be ferromagnetic however, room-temperature magnetization curves for NiTe have been interpreted as ferromagnetic behaviour and have been

compared with studies conducted by others [40,41]. Thus, much contradiction is realized, and therefore, the present data are interpreted keeping such background literature in mind. It is also reported that in the case of NiTe_2 , the dilution in magnetic behavior is caused by Te. However, since the present study is based on Ni-rich NiTe_2 , it is expected that the magnetism will be restored.

Appropriately, in the given plot (Fig. 9), nickel-rich NiTe_2 depicts a more stable magnetic property when tested on a magnetic hysteresis loop. The break in the loop shows that the material holds the magnetic property when magnetism is turned off. The VSM hysteresis loop of the Ni-excess NiTe_2 sample indicates ferromagnetic behaviour with a small but noticeable hysteresis. The magnetization increases gradually with the applied magnetic field, showing that the material responds to the external magnetic field effectively. The presence of a narrow hysteresis loop suggests low coercivity, meaning the material can be magnetized and demagnetized easily, which is characteristic of soft magnetic materials. The non-zero remanent magnetization confirms that some magnetic ordering remains even after removal of the external field. Since the loop is not perfectly linear, the sample does not behave as a purely paramagnetic material.

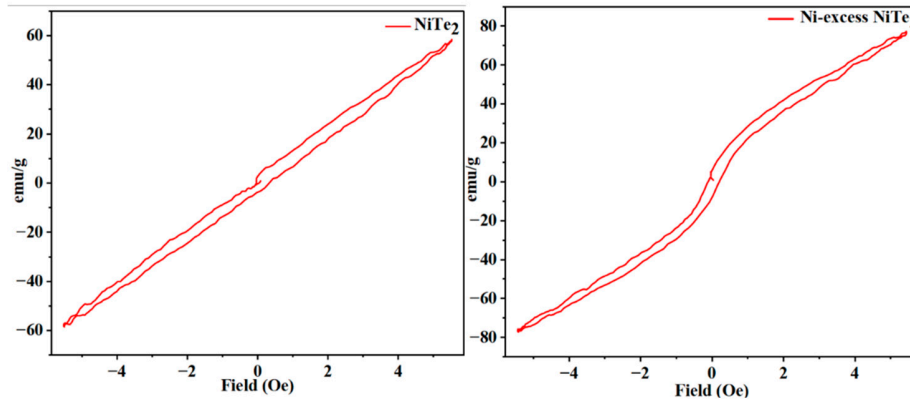


Figure 9: Room temperature magnetic hysteresis curves for NiTe_2 and Ni-rich NiTe_2 showing M-H hysteresis loop with an applied field between -5k and 5k Oe.

3.3 EMI Shielding Studies

EMI shielding studies deal with blocking or weakening of electromagnetic waves by a suitable shield made of material/composite/polymer/metal, etc. In practice, materials usually protect against radiation in two ways, either by reflecting the waves back or by absorbing them within their structure. Materials with moderate electrical conductivity (around 10^{-3} to 1S cm^{-1}) are already capable of shielding by reflection, as their electrons and holes generate ohmic losses that weaken the incoming waves [27–29]. On the other hand, effective absorption requires the electric or magnetic dipoles within the material to interact strongly with the electromagnetic field. EMI performance is evaluated using a vector network analyzer (VNA), which measures the S-parameters, S_{11} (representing how much of the wave is reflected) and S_{12} (representing how much passes through). These values directly relate to reflectance ($R = |S_{11}|^2$) and transmittance ($T = |S_{12}|^2$) [29,31,32].

$$R = |S_{11}|^2 = |S_{22}|^2 \quad (1)$$

$$T = |S_{12}|^2 = |S_{21}|^2 \quad (2)$$

$$\text{SE}_R = -10 \log (1 - R) = -10 \log (1 - |S_{11}|^2) \quad (3)$$

$$SE_A = -10 \log (T/1 - R) = -10 \log (|S_{12}|^2/(1 - |S_{11}|^2)) \quad (4)$$

and

$$SE_T = SE_R + SE_A \quad (5)$$

From these, the absorbance (A) can be calculated using $A = 1 - R - T$. Because part of the incoming wave is always reflected at the surface, the internal absorption is often better understood through the effective absorbance. This approach helps separate the true absorption occurring inside the material from simple surface reflection, giving a clearer picture of how well the material actually dissipates electromagnetic energy [33,34].

$$A_{\text{eff}} = (1 - R - T)/(1 - R)$$

In the present study, NiTe₂/PVA composite films were evaluated for the X-band (8–12 GHz) energy region. The values of SE_R and SE_A are estimated and presented in Tables 2 and 3 (Figs. 10–12). Appropriately, it is observed that the composite film was a much more active EMI material and showed better performance with increasing filler content. Tables 2 and 3 show findings where the samples had different concentrations (w/w) in the PVA matrix and resulted in mild shielding efficiency. The X-band measurements were done using a WR-90 rectangular waveguide in the frequency range of 8.2–12.4 GHz. Typical waveguide dimensions were 0.9 × 0.4 inch (22.86 × 10.16 mm). The samples were prepared of the same dimensions with a thickness of approximately 1.0 mm.

Table 2: EMI SE of NiTe₂/PVA films.

Sample Code	NiTe ₂ (g)	PVA (g)	% Fillers	Film Thickness	SE (R)	SE (A)	EMI SE _T (dB)
DBF-1	0.1	2.0	5%	1.0 mm	0.71	1.56	2.27
DBF-2	0.2	2.0	10%	1.0 mm	1.16	1.36	2.52
DBF-3	0.3	2.0	15%	1.0 mm	0.91	2.16	3.07

It is opined that a higher amount of nickel telluride improves the SE marginally, and an increasing trend has been noticed irrespective of type of nickel telluride. Thus, even the change in material type with respect to crystal structure and stoichiometry, i.e., NiTe₂ versus Ni₂Te₃, offers only marginal advantage in terms of SE. However, surprisingly silver-containing samples yielded lower SE despite their conducting behavior. It is further realized that the absorption component SE(A) was found to contribute more significantly than reflection SE(R) in most samples, indicating that electromagnetic wave attenuation mainly occurs through dielectric loss, interfacial polarization, and multiple scattering within the nanostructured nickel telluride(s). Relatively lower EM reflection suggests that the material behaves more as an absorbing shield rather than a reflecting barrier. Generally, lightweight shields are desired for X and Ku band applications thus, polymers are often the natural choice and have been considered as a highly suitable matrix. Often the thermal and electrical properties of polymers can be tailored by incorporating inorganic fillers. Thus, either the polymer with metal chalcogenides or metal and metal oxides can be utilized for the shielding purpose, or the conducting polymers themselves may yield agreeable results. Indeed, in the present effort, we have tested nickel metal telluride in combination with PVA since nickel telluride has never been studied for EMI applications. Although the current findings suggest low EMI SE, the present exploratory results should

be considered as a novel combination of unexplored material that may have future potential alone or as hybrid, etc. A typical EMI shielding mechanism can be as shown in Fig. 10.

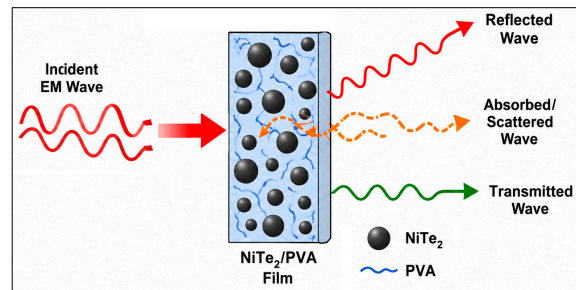


Figure 10: Typical EMI shielding mechanism involving nickel telluride and PVA.

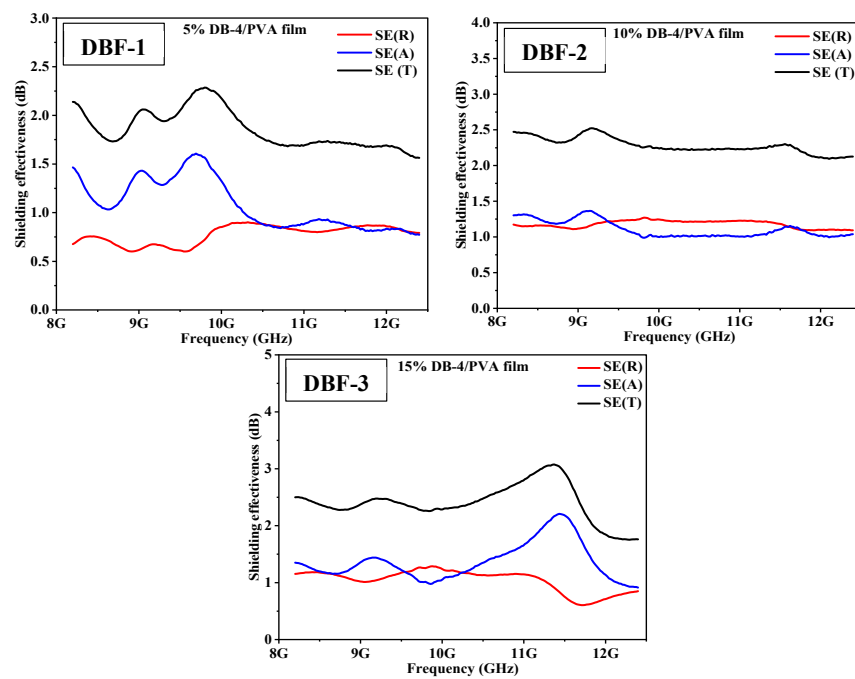


Figure 11: EMI shielding of DB-4 sample based PVA film.

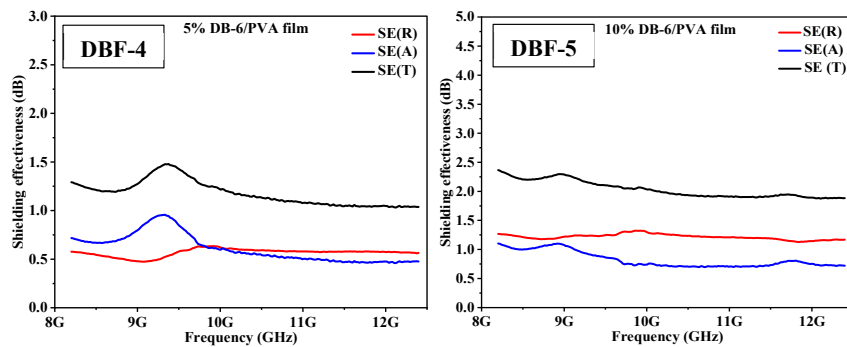


Figure 12: Cont.

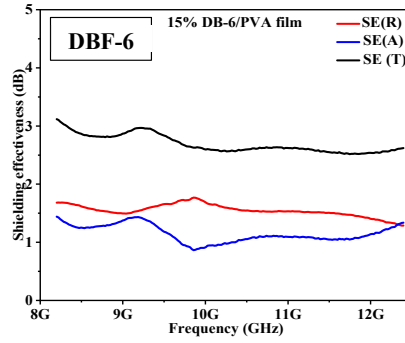


Figure 12: EMI shielding of $\text{Ni}_2\text{Te}_3/\text{PVA}$ film.

Table 3: EMI SE of $\text{Ni}_2\text{Te}_3/\text{PVA}$ films.

Sample Code	Ni_2Te_3 (g)	PVA (g)	% Fillers	Film Thickness	SE (R)	SE (A)	EMI SE _T (dB)
DBF-4	0.1	2.0	5%	1.0 mm	0.52	0.95	1.47
DBF-5	0.2	2.0	10%	1.0 mm	1.26	1.09	2.35
DBF-6	0.3	2.0	15%	1.0 mm	1.67	1.43	3.10

3.4 Thermoelectric Studies

The thermoelectric performance of nickel tellurides was investigated to evaluate its capability as a potential thermoelectric material for energy harvesting applications. NiTe exhibits semiconductor characteristics, good electrical conductivity, and favorable charge transport behavior, which are essential for thermoelectric behavior. Additionally, the presence of tellurium helps reduce thermal conductivity, thereby improving the thermoelectric efficiency of the material [35,36]. Therefore, studying NiTe for thermoelectric generator applications helps determine its potential for converting waste heat into useful electrical energy in sustainable power generation systems (Table 4).

Table 4: Thermoelectric data of nickel telluride/PVA composite films.

S. N.	l (cm)	B (cm)	T (cm)	EC (S/m)	(S) $\mu\text{V/K}$	PF = $S^2\sigma$ ($\mu\text{W/mK}^2$)	TC (k) W/(m·K)	(zT) Unitless
15% NiTe_2 PVA Film	1.0	0.3	0.05	147.1	-156.3	3.59	0.322	0.0033
10% Ni_2Te_3 PVA Film	1.0	0.3	0.05	168.3	-154.8	4.03	0.221	0.0054

l = Length, b = Width, t = Thickness, Electrical conductivity (EC), Seebeck coefficient (S), Power Factor (PF), Thermal Conductivity (TC), Figure of Merit (zT).

The thermoelectric analysis of the synthesized nickel telluride was carried out by converting the powder into PVA composite, and the films were cut to $1\text{ cm} \times 0.3\text{ cm} \times 0.05\text{ cm}$ dimensions. The composite film samples demonstrated moderate electrical conductivity along with negative Seebeck coefficient values, confirming the n-type semiconducting behavior of the material. The electrical conductivity values range from 147.1 for 15% NiTe_2 PVA film to 168.3 S/m for 10% Ni_2Te_3 PVA film, indicating effective charge transport through the interconnected nanostructured network in the matrix. The Seebeck coefficient values marginally vary between -154.8 and -156.3 $\mu\text{V}/^\circ\text{C}$. The negative sign indicates that electrons are the dominant charge carriers in the nickel telluride system. Among the analyzed samples, $\text{Ni}_2\text{Te}_3/\text{PVA}$ film

exhibited comparatively higher electrical conductivity and power factor values despite the lower amount in the film, suggesting improved carrier transport and thermoelectric performance. The calculated power factor values lie in the range of 3.53–4.03 $\mu\text{W}/\text{mK}^2$, with the highest value observed for $\text{Ni}_2\text{Te}_3/\text{PVA}$. The below equation was practiced for the calculation of power factor:

$$\text{Power factor (PF)} = S^2 \sigma \text{ (}\mu\text{W}/\text{mK}^2\text{)}$$

The enhanced power factor may be attributed to better crystallinity, optimized carrier concentration, and interconnected nanostructure morphology. Overall, the obtained results indicate that the synthesized nickel telluride nanoparticles possess promising thermoelectric characteristics and potential for energy conversion applications.

4 Conclusion

Nickel tellurides are particularly promising materials because their electronic and structural properties can be tuned with ease. The article presented the synthesis and characterization of nickel telluride nanoparticles using chemical synthesis methods. The findings from XRD and TEM especially showed excellent material nature and crystallinity as well as directed morphology. The estimated particle diameter from XRD peak width was between 8–14 nm with lattice spacings of 0.29 nm. The ferromagnetic behaviour was revealed by a magnetic property study. Crystal phases such as NiTe , NiTe_2 , and Ni_2Te_3 naturally form layered structures that support fast electron movement, and their bandgap tunability allows them to shift between metallic and semiconducting behaviour depending on particle size and synthesis conditions. Their Ni and Te-rich surfaces further enhance fast electron transfer, strengthening their performance in electromagnetic applications. This flexibility makes them excellent candidates for electromagnetic shielding, where rapid charge redistribution is essential for blocking or absorbing electromagnetic radiation. The study confirms its potential with an in-depth study of the material. Nickel telluride(s) showed that the semiconductor property of the compound plays a key role in shielding effects from electromagnetic radiation and thermoelectric property. The EMI shielding efficiency, although mild, can be enhanced by tuning the material's stoichiometry. The thermoelectric behaviour of nickel telluride also showed a good power factor and can be tailored with appropriate fine-tuning. The composite film samples demonstrated moderate electrical conductivity along with negative Seebeck coefficient values, confirming the n-type semiconducting behavior of the material. The calculated power factor values lie in the range of 3.53–4.03 $\mu\text{W}/\text{mK}^2$, with the highest value observed for $\text{Ni}_2\text{Te}_3/\text{PVA}$.

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Conflicts of Interest: The authors declare no conflicts of interest.

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