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Access to lanthanoid telluride nanoparticles: Liquid exfoliation of $LnTe_3$ (Ln = La, Ho)



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ABSTRACT

Although many of the nanoparticles containing lanthanoids are deeply investigated, the reports concerning chalcogenide materials are scarce. Comprehensive exploration of properties of these materials requires a proper preparation method avoiding hydrolysis and contamination with oxygen-containing products. Hence, in this case, the top-down approach could be preferable. In this study we demonstrated preparation of LnTe₃ (Ln = La, Ho) nanoparticles by liquid exfoliation method. Bulk tritellurides LaTe₃ and HoTe₃ afford stable dispersions in a number of common organic solvents under ultrasonic treatment. The size and morphology of nanoparticles in colloid solutions as well as films prepared from them were investigated by a number of methods (AFM, TEM, DLS, XRD, Raman). LnTe₃ form stable dispersions in alcoholic media with concentration up to 165 mg/L. In these dispersions nanoparticles exist as 1–2 layered charged flakes with lateral size 50–80 nm. The nanoparticles can be reassembled as films, which demonstrated orientation along 0*k*0 direction.

1. Introduction

A great deal of attention has been focused on synthesis and characterization of 2D-materials, especially after discovering of graphene and its fascinating properties [1]. One of the most important families of 2D materials is the chalcogenide compounds with layered structure such as MoS₂, WS₂ [2,3], Bi₂Te₃ [4], NbS₃ [5], etc. Due to exotic electronic properties and high surface areas these materials are promising for catalysis [6,7], sensing [8,9], energy storage [10], and other applications.

A significant attention has been focused on the synthesis and characterization of 2D-materials, especially after discovery of graphene and its fascinating properties [1]. One of the most important families of 2D materials is the chalcogenide compounds with layered structure such as MoS₂, WS₂ [2,3], Bi₂Te₃ [4], NbS₃ [5], etc. Due to exotic electronic properties and high surface areas these materials are promising for catalysis [6,7], sensing [8,9], energy storage [10], and other applications.

Among chalcogenides of different metals those of lanthanoid (Ln)

elements demonstrate potential as high-temperature thermoelectric materials [11,12], solar energy conversion materials [13], pigments [14], infrared window materials [15], and phosphor host media [16]. Because of the high oxophility of lanthanoids synthesis of such nanoparticles remains a challenging problem. Conventional methods applied to other lanthanoid nanoparticles (sol-gel, spray-pyrolysis, hydrothermal, etc.) in case of chalcogenides can result in hydrolysis to oxygen-containing products. According to hard soft acid base theory (HSAB) [17], for hard ion Ln^{3+} it is more preferable to make bonds with hard ions (O^{2-} , F^-) than with much softer Q^{2-} ($Q^{2-} = S^{2-}$, Se^{2-} Te^{2-}). While a large body of works about preparation, properties and application of various lanthanoid compounds were reported (e.g. nanorare-earth oxides [18-21] and fluorides [22-24]) the number of studies describing nanostructured Ln chalcogenides are scarce. Preparation of EuS and EuSe was previously reported by a number of researchers [25-27]. There are only several studies of trivalent Ln sulfides [28-30] and a few examples of the selenides and tellurides [31-34].

For preparation of nanoparticles and 2D materials, a liquid exfoliation is one of the cheapest, fastest and effective methods [35].

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Abbreviations: TMDC, transition metal dichalcogenides; DLS, dynamic light scattering; iPrOH, isopropyl alcohol; EtOH, ethanol; Py, pyridine; iAOH, isoamyl alcohol; NMF, N-methylformamide; DMF, dimethylformamide; NMP, N-methylpirrolidone

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Fig. 1. Crystal structure of LnTe₃.

During this process, ultrasonic waves are applied to a mixture of bulk layered material and a properly selected solvent, providing enough energy to overcome the attraction forces between layers and generating exfoliated nanosheets. After sonication, the dispersion is centrifuged to remove particles that were not exfoliated. The result is a supernatant containing a colloidal suspension of material only a few layers thick. This approach was widely used for different layered materials with van der Waals gap in the structure as, e.g., transition metal dichalcogenides (TMDC) [36], BN [37] and graphene [38].

Among rare earth binary chalcogenides only LnQ_{2-x} (Q = S, Se, Te), Ln_2Te_5 and $LnTe_3$ possess layered structure. The latter compounds crystallize either in *Cmcm* or distorted *Ama2* orthorhombic structure composed of double layers of planar Te sheets separated by corrugated Ln_2Te_2 layers which are bonded by weak van der Waals interactions (Fig. 1). Such structure suggests that $LnTe_3$ can be exfoliated under appropriate conditions similarly to the TMDCs. Due to their structure and associated specific electronic structure, $LnTe_3$ has lately received significant attention as the first system in which nominal square-planar symmetry is broken by the formation of a unidirectional charge-density wave resulting in satellite reflections in diffraction characterization [39]. It is of great interest to examine these materials when the thickness of the crystal is limited by several nanometers.

To demonstrate applicability of this approach to different Ln, for our investigation we selected one light (cerium group) and one heavy (yttrium group) lanthanoids crystallizing in different space groups (Cmcm or Ama2 respectively). The latter structure exhibits elongation of the Ln-Te bond and a slight distortion of Te sheets while preserving the overall structure motif. In this report we present results of liquid exfoliation of LaTe₃ and HoTe₃ in different solvents as well as the study of their colloidal dispersions obtained by ultrasonication.

2. Materials and methods

2.1. Synthesis of LnTe₃

Compounds $LnTe_3$ (Ln = La, Ho) were prepared from stoichiometric mixtures of high-purity Ln and Te, with I₂ as mineralizer [40]. Samples were kept under inert atmosphere, as these compounds show noticeable change after several days of exposure to air.

0.472 g (3.4 mmol) of lanthanum and 1.302 g (10.2 mmol) of tellurium or 0.398 g (2.4 mmol) of holmium and 0.924 g (7.2 mmol) of tellurium was placed into a quartz tube. The tube was evacuated and filled with argon (repeated 3 times). 0.004 g (1.5×10^{-2} mmol) of iodine was placed into the tube filled with argon, then it was cooled in liquid nitrogen, evacuated and sealed. The tube was heated to 700 °C and held at this temperature for 72 h, then slowly (5 °C/h) cooled to 540 °C, after the heating was turned off. In both cases bronze-colored microcrystalline powders were obtained after reaction completion. The yields were quantitative: 1.772 g (99%) for LaTe₃ and 1.320 g (99%) for HoTe₃. There was no iodine in the obtained products according to EDS analysis.

2.2. Suspension preparation

Solvents were dried before use to remove residual water and to prevent possible hydrolysis of rare-earth telluride nanoparticles. Isopropanol (iPrOH) was distilled over Mg, acetonitrile over P2O5. ethanol (EtOH) over Na, pyridine (Py) over KOH, dimethylformamide (DMF) over BaO under low pressure and N-methyl pyrrolidone (NMP) over Na₂SO₄ under low pressure. Acetone was kept over molecular sieves 4 Å. Other solvents (isoamyl alcohol (iAOH), N-methylformamide (NMF)) were used as purchased. All solvents were deoxygenated prior to use by heating-cooling combined with argon bubbling. In a typical procedure of preparation of colloidal suspensions 10 mg of LnTe₃ and 10 mL of solvent was placed in glass flask, then again argon gas was bubbled through the liquid for 10 min, after that the flask was closed and sonicated using ultrasonic bath Sapphire (ultrasonic power 150 W, frequency 35 kHz) for 8 h in three times with a gap of 16 h. During ultrasonication, samples were termostated at 20-22 °C to prevent thermal restacking of colloidal particles. Resulting mixture was centrifuged for 10 min at 3000 rpm to sediment large particles, and upper 4/5 of supernatant solution were taken for further analysis. The colloidal dispersions were black or dark violet-brown at low concentration.

2.3. Preparation of films of colloidal dispersions

Films of the $LnTe_3$ were prepared to study their Raman spectra and X-ray diffraction. For this purpose 100–200 mL of the dispersion was filtered under low pressure through 'Vladipor' membrane filter with pore size of 50 nm.

2.4. Characterization

The X-ray powder diffraction (XRD) analysis of the samples was performed on a Shimadzu XRD7000 diffractometer (CuK α radiation, Ni filter, $2\theta = 10^{\circ}$ –70°). Raman spectra were recorded with a Spex Triplemate spectrometer. UV spectra were recorded with UV-3101 PC Shimadzu spectrometer in the range 200–800 nm. The infrared (IR) spectroscopy performed on a Scimitar FTS 2000 IR Fourier spectrometer. The samples were prepared as suspensions of powdered films in Nujol medium and measured between KBr windows. The DLS measurements were carried out in a 1 cm glass cuvette in a spectrometer NanoBrook Omni (Brookhaven Inst., USA). Scattering angle was 90°, for each measurement photon accumulation time was 10 s, the temperature was 20 °C (accuracy 0.1 °C). Autocorrelation was performed by spectrometer software with cumulant method for monomodal analysis and

the NNLS (non-negatively constrained least squares) algorithm for polymodal analysis. Z-averaged (by intensity from 60 to 100 measurements) hydrodynamic diameter Dhz was calculated by Stokes-Einstein equation [41] for spherical particles. For in-depth investigation of the systems, the n-averaged (by number) hydrodynamic diameters Dhn were calculated by monomodal and polymodal analyses. Electrokinetic potentials (ζ -potential) of the particles in solutions were recorded with spectrometer NanoBrook Omni (Brookhaven Inst., USA) by laser electrophoresis method with phase analysis light scattering (PALS). Photons scattered by the particles were detected at the angle 15°. Measurements were carried out at 25 °C in special cell SRR2 resistant to organic solutions (Brookhaven, USA) equipped by parallel plate palladium electrodes with surface $\approx 45 \text{ mm}^2$; a gap between electrodes was 3.45 mm. Measurements were carried out in the manual mode, ζ -potential were determined by Smoluchowski model from 10 to 20 measurements. The solutions were measured without dedusting. The error did not exceed 5%. The scanning microscope Solver PRO NT-MDT was used to obtain AFM images of the film surfaces, to determine their thickness and to study the structure. AFM measurements were performed in the semi-contact mode. Images were edited and analyzed using Image Analysis 3.5.0.2069 and ImageJ software [42]. The samples were prepared using several drops of diluted colloidal dispersions, which were applied to silicone pieces spinning at 1000 rpm and dried at the ambient temperature. HRTEM micrographs were obtained by Cscorrected transmission electron microscope Jeol JEM- 2200FS with point-image resolution 1.9 Å and acceleration voltage of 200 kV. Analysis of the local elemental composition (atomic %) was carried out using energy-dispersive EDX spectrometer. Concentration of the dispersions was determined by Thremo Scientific iCAP-6500 ICP-AES spectrometer. For this analysis several milliliters (1-3 mL depending on the initial concentration) of dispersion were dissolved in aqua regia (HCl : $HNO_3 = 3:1$), and then diluted with water to 50 mL, the samples being analyzed by AES for lanthanum or holmium. Contact angles were measured by Drop Shape Analyzer DSA-100E, KRÜSS. Analysis of the contact angles of droplet on samples by height-width method processed in ADVANCE software (KRÜSS), volume of droplet was 2 µL. Surface free energy was calculated using Owens, Wendt, Rabel and Kaelble method (OWRK) with two liquids: distilled water (H2O) as polar, and diiodomethane (CH₂I₂) as non-polar one.

3. Results and discussion

3.1. Solvents survey

According to reported data for exfoliated colloidal systems, the solvent plays the key role in stabilizing dispersions and therefore, their concentration. Initially we choose available solvents, which have been found to be effective for exfoliation of TMDC and KLnS₂ [43]. Hydrolysis occurs immediately in the case of H₂O, and even traces of water in common solvents significantly decrease the stability of the dispersions, that is why we had to distill them over desiccants. UV-vis spectra (Fig. 2a) are often used for estimation of the concentration of dispersions by the Lambert Beer law: A/l relates to the concentration as A/ $l = \alpha C$, where α is the extinction coefficient, A is absorbance at certain wavelength, *l* is cell length, and *C* is the concentration. In our case, we found that characteristic bands (256 nm for HoTe₃ and 275 nm for LaTe₃) of the tellurides lay in the region where many of the solvents have considerable absorbance, so it is unlikely that the extinction coefficient should be the same for all used solvents. We evaluated the concentrations by ICP-AES method instead. The experimental error did not exceed 10%. The measurements of the concentrations were performed after centrifugation to sediment the large (heavy) particles. The results are shown in Table 1. For iPrOH and EtOH dispersions we also calculated extinction coefficients and found that their values were different (Fig. 2b); it means that scattering component of the coefficient wasn't constant for each material and solvent. Most stable and

concentrated dispersions were achieved in isopropanol (iPrOH) and ethanol (EtOH), whereas acetonitrile and acetone tended to form unstable dispersions (almost all dispersed material settled after centrifugation). N-methylformamide slowly reacted with the tellurides resulting in a change of color of the substance. No stable dispersion was obtained in this case.

It is known that the concentration of the dispersion is maximized when the surface energies of the particle and the solvent match. This energy can be roughly estimated through surface tension or more precisely using Hildebrand or Hansen solubility parameters [3], and inverse gas chromatography [44,45]. Taking into account the important role played by surface energy in the exfoliation of layered materials, we believe it essential to estimate of the actual surface energy of these materials. To do this, we used simple and effective method: contact angles measurements. There are various approaches of determination of the surface free energy from contact angles, i.e. Zisman, Fowkes, Owens-Wendt, Wu, acid-base theory, Neumann etc. In this work we have used Owens Wendt, Rabel and Kaelble method (OWRK), described elsewhere [46-48]. Unfortunately, considerable imbibition of used liquids into the films of the LnTe₃ prepared by filtering of dispersions onto porous alumina membrane prevented the correct measurements of contact angles. These angles were low and changed during time of measurements. Thus, the films were scratched from filter and pressed into pellets.

The measured contact angles for LnTe3 were close to each other: 63.60° (LaTe₃) and 54.59 (HoTe₃) for water and 21.66° (LaTe₃) and 18.02 (HoTe₃) for diiodomethane. The estimated values of dispersive and polar components of the energies were 47 mJ/m^2 and 8 mJ/m^2 respectively for LaTe₃ resulting in 55 mJ/m^2 of total surface energy. For HoTe₃ we found a little higher values of polar component (12 mJ/m^2) with similar dispersive energy value of 48 mJ/m^2 . This implies the total surface energy to be around 60 mJ/m^2 for HoTe₃. The values of dispersive parts of surface energies of LaTe₃ and HoTe₃ are reasonably close to 40-44 mJ/m² measured for MoS₂ and MoSe₂ [45]. Thus it could be possible to exfoliate LaTe₃ in such solvents as different pyrrolidone derivatives (cyclohexylpyrrolidone, N-Vinyl-pyrrolidone, etc.). For NMP our experiments confirmed relatively high concentration of dispersion of LaTe₃. However NMP as a solvent has some disadvantage due to its high boiling point and toxicity, therefore we focused our attention on alcoholic dispersions.

3.2. Morphology and sizes of nanoparticles obtained from colloidal dispersions

For examination of the size and morphology of obtained particles several methods were applied. Transmission electron microscopy (TEM) images of LnTe₃ (Fig. 3b) deposited on carbon TEM grid indicated that particles have irregular shape and tend to stick together when solvent evaporates, probably due to high surface tension. They looked like thin nanoplates with lateral size around 50-70 nm along with the presence of larger particles. High-resolution TEM (HRTEM) image showed that the particles consist of small slabs around 10 nm in size staked on a bigger one (Fig. 3b, circled). Fourier-transformed image confirms that these tiny slabs have similar d-spacing but different orientations; as one can see as several spots lay on a ring (Fig. 3b, inset), and these d-spacings can be indexed as (130) and (111) reflections of LaTe₃ particles. Atomic force microscopy observations were carried out to determine the thickness of the particles (Fig. 3c). We performed a statistical analysis on more than 300 particles; results are shown in Fig. 3d. The major part of the particles was 1-2 nm thick, with presence of larger sized agglomerates. In case of HoTe3 the distribution of particles' thickness was narrower, but the maximum also was at 1-2 nm. Aggregation observable in the TEM image took place in AFM images too. According to the crystal structure, one layer corresponds to 1 nm, thus most of the particles consist of two layers.

Z-averaged hydrodynamic diameters D_{hz} , measured by dynamic



Fig. 2. UV-vis spectra of LnTe₃/iPrOH and Ln/EtOH dispersions (a). Calibration line for determination of extinction coefficient of the LnTe₃/iPrOH and LnTe₃/EtOH dispersions.

 Table 1

 Concentration of the dispersions in different solvents.

Solvent	$LaTe_3$ concentration, mg/L	HoTe $_3$ concentration, mg/L
iPrOH	80	65
EtOH	110	165
Ру	50	80
iAOH	30	40
Acetonitrile	Poor dispersion	Poor dispersion
Acetone	Poor dispersion	Poor dispersion
NMP	80	20
DMF	30	110
NMF	No dispersion	No dispersion

scattering (DLS) method in iPrOH and EtOH dispersions were 187 and 248 nm for LaTe₃ and 195 and 210 for HoTe₃ respectively. The lognormal monomodal particles size distribution for the LaTe₃/EtOH dispersion is shown in Fig. 3e. It should be noted that this parameter was calculated in assumption of the spherical shape of the particles and averaging by intensity (z-averaging) describes better the particles with larger diameters, because they make the main contribution into light scattering.

For advanced examination of the colloidal solutions, we performed a polymodal analysis of DLS. In case of z-averaged diameter D_{hz} it gives bimodal distribution of for all investigated systems (Table 2). For naveraged (by number, D_{hn}) diameters the larger mode disappeared, indicating that amount of bigger particles (> 100 nm) is insignificant. Keeping in mind that using the n-averaged diameters is essential for



Fig. 3. TEM (a), HRTEM (b) and AFM (c) image of LaTe₃/iPrOH nanoplates. Distribution functions of particles thickness from AFM images for LnTe₃ nanoplates. DLS Lognormal particles size distribution for LaTe₃/iPrOH (d).

Table 2

DLS measurements and zeta-potential for EtOH and iPrOH dispersions.

LnTe ₃ /solvent	DLS, monomodal analysis	DLS, polymodal analysis				Calculated disc diameter ^{*,} d, nm	Zeta-potential
	D _{hz} , nm	D _{hz} , nm		D _{hn} , nm		d, nm	ζ, mV
		Mode 1	Mode 2	Mode 1	Mode 2		
LaTe ₃ /EtOH	248	61	348	57	-	75	-26.7
LaTe ₃ /iPrOH	187	46	271	45	-	59	36.9
HoTe ₃ /EtOH	210	61	292	61	-	80	-34.0
HoTe ₃ /iPrOH	195	52	274	51	-	67	45.3

* Calculation from D_{hn} by Eq. (1) for h = 2 nm (AFM).



Fig. 4. Powder diffraction patterns: 1 – LnTe₃/EtOH film, 2 – LnTe₃/iPrOH film, 3 – Bulk LnTe₃ 4 – simulation of oriented in 0k0 direction LnTe₃ powder for HoTe₃ (black) and LaTe₃ (red) (a). Raman spectra of LaTe₃ (b) and HoTe₃ (c) films. IR spectra of LnTe₃ powder after ultrasonication (d). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

comparison with TEM data, we use D_{hn} in further examination of particles' sizes and stability of dispersions.

From AFM and TEM observations the particles have platelet-like shape with the thickness remarkably smaller than the lateral size. It seems reasonable to approximate shapes of the particles as discs with the diameter d and thickness h. Assuming the parameter h is equal 2 nm from AFM data, diameter d could be calculated using the equation [49]:

$$D_{hn} = \frac{3}{2}d\left[\left[1 + \left(\frac{h}{d}\right)^{2}\right]^{\frac{1}{2}} + \frac{d}{h}\ln\left[\frac{h}{d} + \left[1 + \left(\frac{h}{d}\right)^{2}\right]^{\frac{1}{2}}\right] - \frac{h}{d}\right]^{-1}$$
(1)

Obtained values *d* (Table 2) fall within range of 60–80 nm, being in good agreement with TEM data.

The studied dispersions are stable for a long time (up to several months), and aggregated precipitate can be easily re-dispersed by short sonication. Determination of the ζ -potential performed in iPrOH and EtOH dispersion showed that nanoparticles in isopropanol had the

positive charge and in ethanol the negative charge (Table 2); however, the origin of these charges is unknown. Perhaps, they generated on the edges of particles due to disruption of some Ln-Te bonds during ultasonication. The relatively high values of zeta-potential (around 30 mV) are in agreement with the observed stability.

3.3. PXRD and Raman spectra of the LnTe₃ films

According to PXRD pattern (Fig. 4a), all of the peaks of the sample can be attributed to $LnTe_3$ and no possible products of hydrolysis were found. PXRD pattern demonstrated strong texture along the *b* direction – the layers in the crystal structure are packed along this direction as well. Reflections 0k0 were narrow and enhanced in intensity, while reflection from other crystallographic planes (Fig. 4a), have considerable broadening compared to 0k0, indicating the specific nature of stacking of the nanoplates in the films.

Raman Spectra of both initial compounds and the films deposited by filtration contain two bands in region $110-150 \text{ cm}^{-1}$ (Fig. 4b, c, insets) The features of spectra appeared to be the same as for HoTe₃ and LaTe₃. Noticeable shift in band positions is observed for the films prepared from EtOH dispersions. There is also some broadening of all the bands, which confirms the nanosized nature of the compounds. We suppose that some interaction between the nanoplates and solvents exist, but as can be seen from Raman and IR spectra (Fig. 4b, c, e), there are no additional bands in 400–1500 cm⁻¹ and 1500–4000 cm⁻¹ region of Raman and IR spectra, respectively. The lack of vibrations in the "fingerprint" region of solvents indicates that, even if some solvation (or solvolysis) could occur during ultrasonication, it is reversible.

4. Conclusion

For the first time we demonstrated the preparation of the telluride lanthanoids nanoplates using liquid exfoliation method. Bulk $LnTe_3$ could be transferred into colloids by ultrasonic treatment in several solvents such as isopropanol, ethanol and pyridine. The most effective solvent is ethanol in which concentration of the lanthanoid compound reaches 165 mg/L. In the dispersions $LnTe_3$ exists as charged nanoplates, and despite some stacking, the thickness of the particles is 1–2 layer with lateral size 50–80 nm. Nanoparticles can be assembled in films by filtering.

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