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Sustainable liquid-phase exfoliation of layered materials with non-toxic solvent

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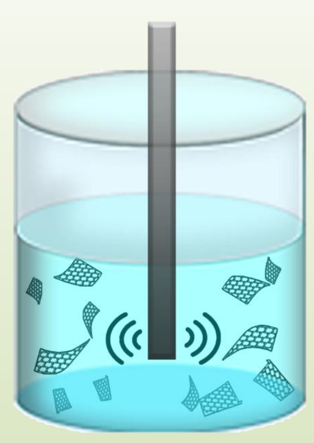
Introduction

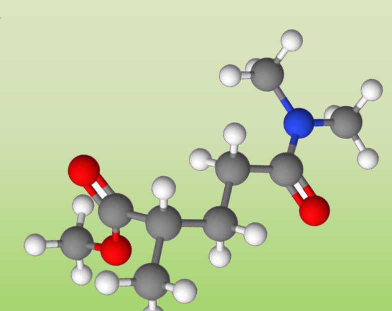
Liquid-phase exfoliation (LPE) is the most suitable platform for large-scale production of two-dimensional materials. One of the main open challenges is related to the quest of green and bio-derived solvents to replace state-of-the-art dispersion media, which suffer several toxicity issues.

Here, we demonstrate the suitability of methyl-5-(dimethylamino)-2-methyl-5-oxopentanoate (Rhodiasolv®Polarclean) for sonication-assisted liquid-phase exfoliation of layered materials for the case-study examples of WS_2, MoS_2 and graphene, given its compatibility in terms of physical properties and its *green* nature.

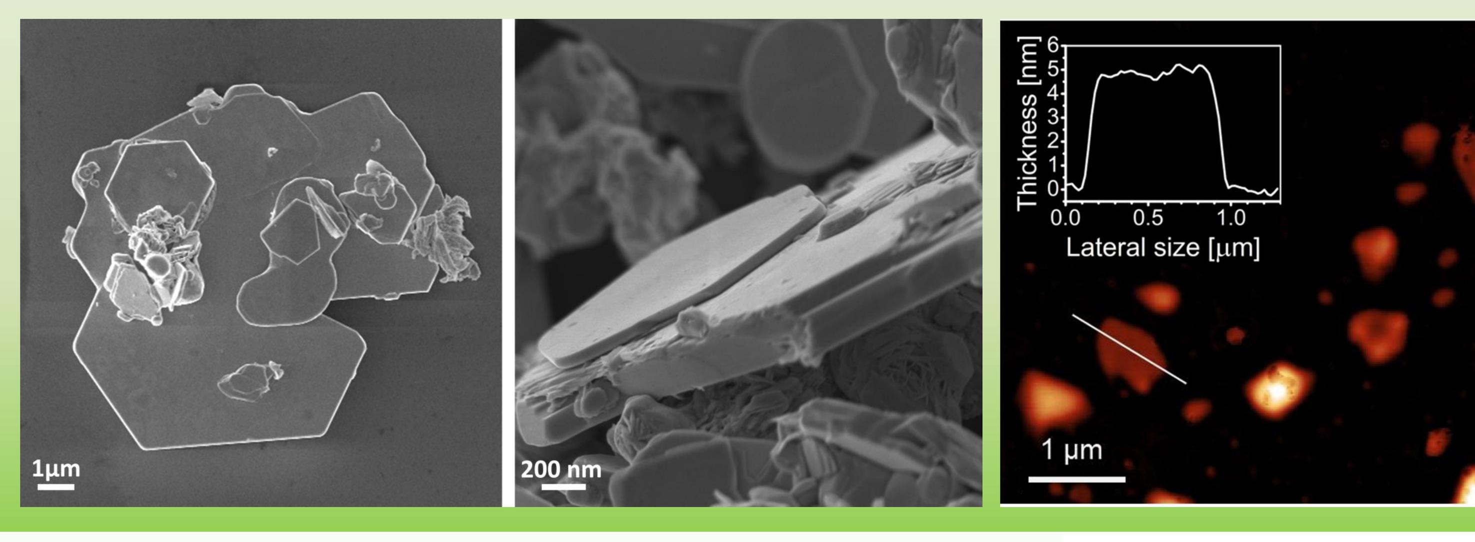
Results

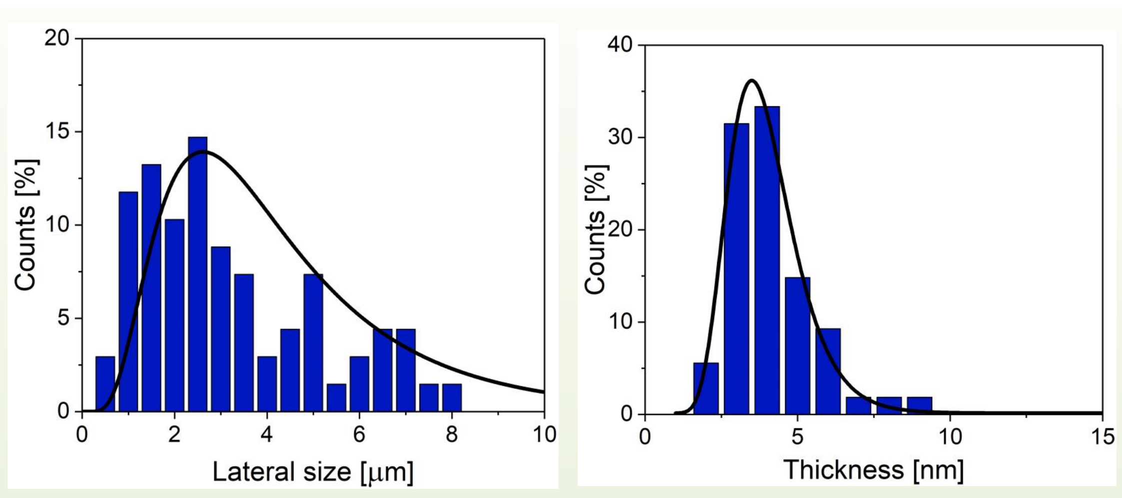
WS_2 powder was dispersed in 40 mL of solvent and sonicated for 3 h in bath sonicator in a thermostat bath to prevent excessive temperature rise (T≤25 °C). In order to physically remove the solvent, several centrifuges were carried out.



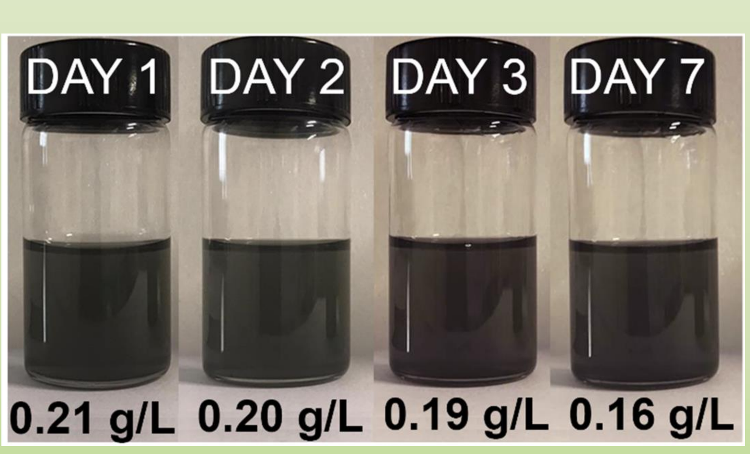


The morphological characterization of exfoliated WS_2 flakes was carried out by means of SEM and AFM microscopy. The images reveal the occurrence of flakes with different lateral sizes generally larger than 1 µm and well-defined hexagonal edges at around 120°. The representative AFM image with the corresponding height profile collected along the with line, allow concluding that Polarclean-assisted LPE provides flakes with an aspect ratio of ~10^3.



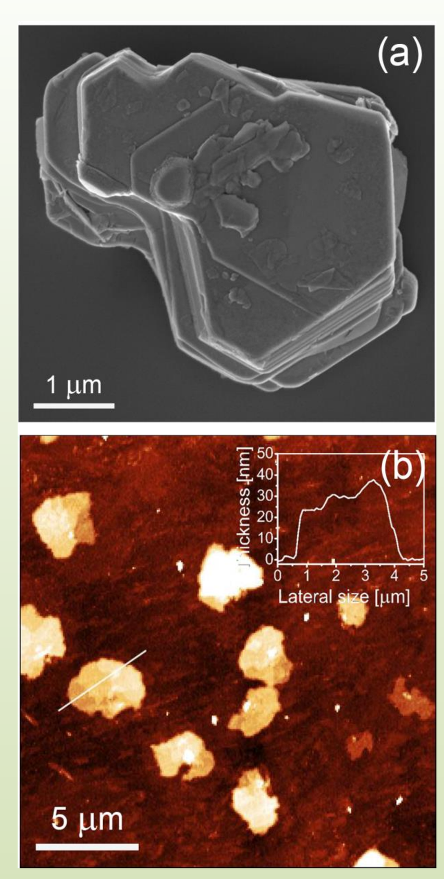


Statistical analysis of lateral size and thickness of WS_2 flakes based on SEM and AFM images demonstrate that lateral size and thickness of the flakes approximately follow log-normal distribution peaked at ~3 µm and ~4 nm respectively.

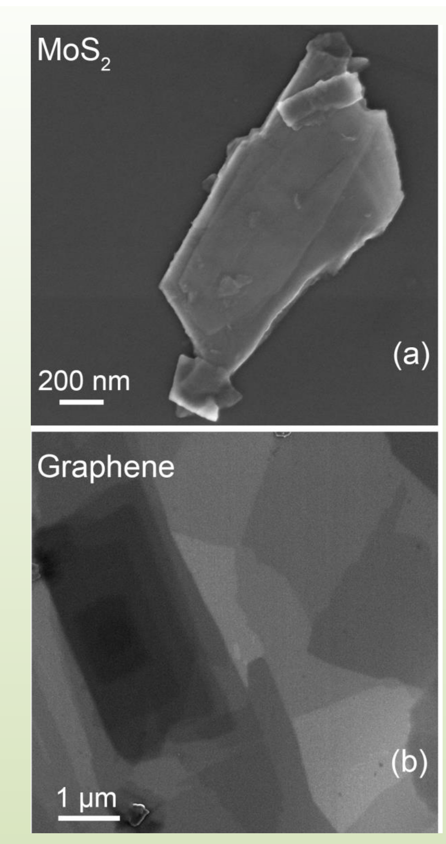


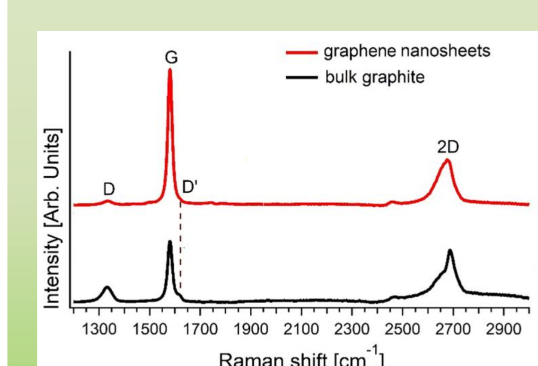
The efficiency of Polarclean for obtaining high-yield and stable dispersions of flakes of 2D materials was validated by means of the analysis of dispersed flakes for the case-study example of WS_2.

Direct comparison, carried out in the same operating conditions, with LPE using N-methyl-2-pyrrolidone (NMP) solvent revealed that the yield of few-layers flakes (with thickness <5 nm) in dispersions obtained by using Polarclean is increased by ~350% as compared to the case of liquid-phase exfoliation performed with NMP, maintaining comparable values of the average lateral size.



The procedure was extended also to MoS_2 and graphene. Regarding MoS_2, statistics reveal results comparable with that of WS_2. For graphene, remarkably the distribution of lateral size shows an average value of 10 µm, which is one of the largest ever reported for LPE.





Moreover, the I_D/I_G ratio as low as 0.07±0.01 in graphene Raman spectra evidences the very low amount of defect induced by exfoliation.

Conclusions

Our results indicate that Polarclean represents a green candidate solvent for large-scale and scalable production of functional inks based on 2D materials, which naturally enables expanding the use of 2D materials in several application fields, for which state-of-the-art solvents have represented so far serious obstacles, owing to their toxicity.

References

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