

Synthesis and Characterization of MoSe₂ Nanoscrolls via Pulsed Laser Ablation in Deep Eutectic Solvents. Supplemental Information.

Figure S-1. Size distribution of MoSe₂

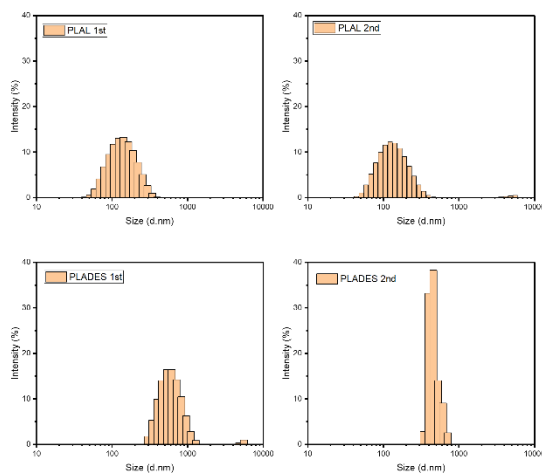


Figure S 1. Size distribution of MoSe₂ in DI water (PLAL) and reline (PLADES)

Size distribution of MoSe₂ shows preference for larger sizes in material ablated in deep eutectic solvent, and also show decreasing size with secondary irradiation. The PLAL samples passes from mono to bimodal distribution. In contrast PLADES synthesized sample passes from bimodal to mono disperse distribution.

Figure S-2. ¹HNMR of MoSe₂ produced in reline

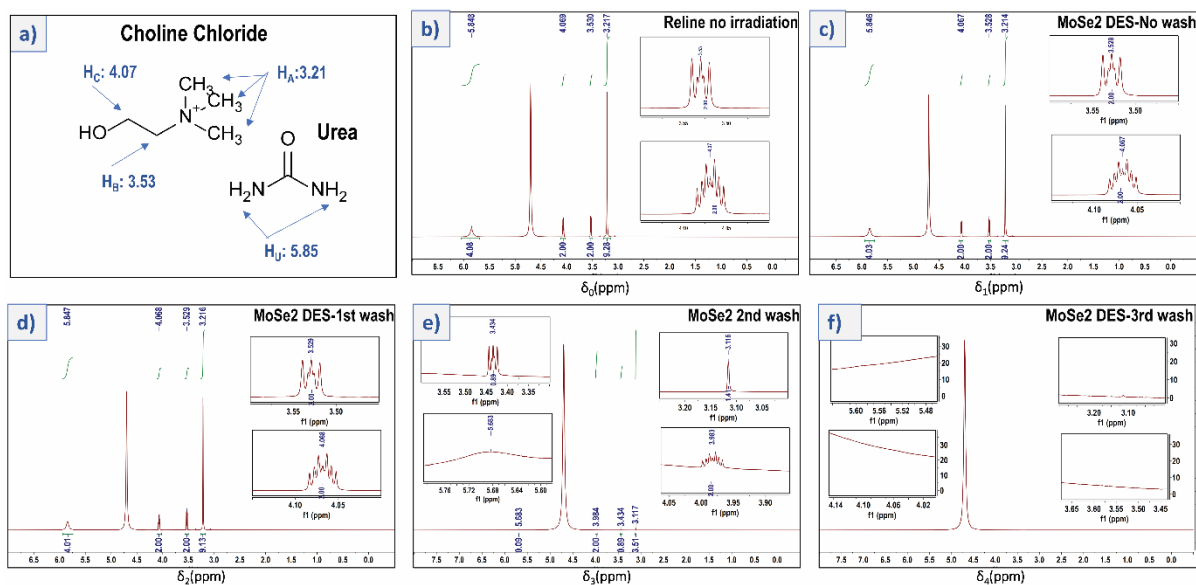


Figure S 2. ¹HNMR results of the serial waching steps suggesting no change in solvent composition by laser irradiation.

The experimental chemical shift of reline was measured before near infrared (NIR) irradiation figure S-2.a. No change in resonance frequency of reline was identified. The chemical shift of Reline after irradiation of MoSe₂ target (figure S-2.c), and after 1st, (figure S-2.d), 2nd (figure S-2.e) and 3rd (figure S-2.f) washing steps, were compared to the untreated neat reline (figure S-2.b). Chemical shift of Reline before and after irradiation conserve the same values and relative abundance, suggesting that there is no formation of secondary species due to solvent degradation by the interaction of 1064 nm pulsed laser.

Table TS-1. ¹HNMR of MoSe₂ produced in reline

Label	Treatment	Washed	H _A	H _B	H _C	H _U
δ ₀	Neat reline, no irradiation	No	3.22	3.53	4.07	5.85
δ ₁	Reline +MoSe ₂ NIR laser	No	3.21	3.53	4.07	5.85
δ ₂	Reline +MoSe ₂ NIR laser	1X	3.22	3.53	4.07	5.85
δ ₃	Reline +MoSe ₂ NIR laser	2X	3.12	3.43	3.98	5.68
δ ₄	Reline +MoSe ₂ NIR laser	3X	--	--	--	--

Table TS 1. Chemical shift of deep eutectic solvent before and after ablation, and after removing reline

The chemical shift remains in the same range as neat reline. Shift upfield after second washing step suggest formation of hydrogen bonding with water of the remaining DES molecules, thus increasing the proton shielding.

Figure S-3. X-ray photoelectron spectrum after edging

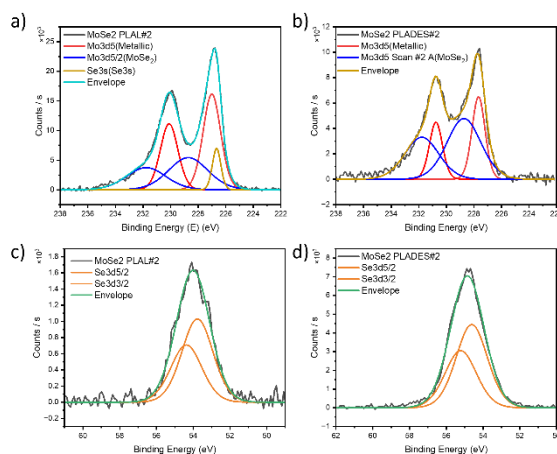


Figure S 3. X-ray photoelectron spectra of MoSe₂ after edging

After 20 seconds of edging, within the Mo3d region, PLAL synthesized MoSe₂ structures show presence of Mo⁺⁶ (metallic Mo) at 227.1 eV in large quantities and small amount of MoSe₂ peaking at 228.7 eV (figure S-3.a). In contrast PLADES sample has large peaks of MoSe₂ at 228.8eV in comparison to the metallic Mo⁺⁶ at 227.7 eV (figure S-2.b). Se3d region of PLAL treatment shows a 5/2 spin orbit split at 53.81 eV (figure S-2.c), while PLADES same feature peaks at 54.65 eV (figure S-2.d). These results suggest lower oxidation during synthesis in deep eutectic solvent, beyond the exposed surface.

Figure S-4. MoSe₂ monolayer Se-Se distances

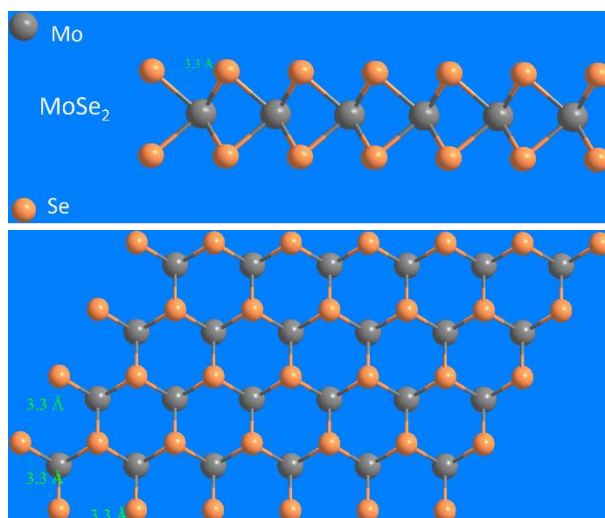


Figure S 4. MoSe₂ monolayer with a interplanar Se-Se distance of 3.3 Å

Representation of the rhombohedral MoSe₂ monolayer obtained by pulsed laser ablation in deep eutectic solvent corresponding to TEM and DP analysis, displaying distances between adjacent selenium atoms in the same layer (created using Vesta and ChemDraw 3D).

Figure S-4. Measured Se-Se distances of MoSe₂ layer synthesized by PLADES

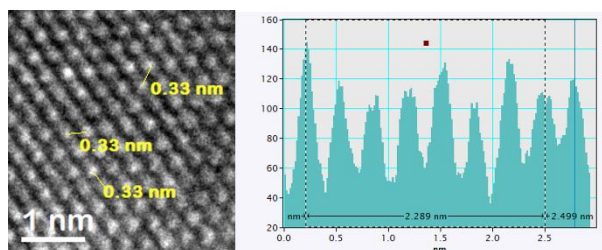


Figure S 5. MoSe₂ nanosheets with inter atomic distances of Se-Se 3.3 Å, analyzed by FFT and line profile.

HR-TEM micrographs of MoSe₂ nanosheets were analysed with line profile at atomic resolution, where recorded distances in between Se-Se atoms on the surface layer is 3.3 in all three directions corresponding to the 1T crystal structure.