



Research papers

Experimental insights of solvent polymeric matrix and electric field influence on dielectrophoretic assembling of MXene-based composites

Ina Turcan^a, Tudor-Alexandru Filip^{a,b,*}, Tăchiță Vlad-Bubulac^c, Daniela Rusu^d,
Marius Andrei Olariu^{a,b}

^a "Ion Ionescu de la Brad" Iasi University of Life Sciences, 3 Mihail Sadoveanu Alley, Iasi 700490, Romania

^b Department of Electrical Measurements and Materials., Faculty of Electrical Engineering, Gheorghe Asachi Technical University of Iasi, 21-23 Professor Dimitrie Mangeron Blvd, Iasi 700050, Romania

^c Department of Polycondensation and Thermally Stable Polymers, "Petru Poni" Institute of Macromolecular Chemistry, 41A Grigore Ghica Voda Alley, Iasi 700487, Romania

^d Department of Physics of Polymers and Polymeric Materials, "Petru Poni" Institute of Macromolecular Chemistry, 41A Grigore Ghica Voda Alley, Iasi 700487, Romania

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ABSTRACT

Harnessing the outstanding properties of MXenes in polymeric-based materials development requires careful consideration of their structural characteristics, as their organization coordinates composite's functional performances. Herein, we investigate the electro-responsive behavior of $Ti_3C_2T_x$ in both polar and non-polar solvents, including polyethylene glycol. Frequency-dependent effect of AC electro-osmotic flow (ACEO) and dielectrophoretic force was assessed, the first one being predominant at lower frequencies in low-conductivity media, while at 10 kHz and above, the diminishing electric double layer (EDL) response suppressed electro-thermal effect (ETE) and enabled positive DEP (pDEP) which governed MXenes motion. Lower polyethylene glycol (PEG) concentrations facilitated MXenes rapid assembling as chains, while higher PEG levels suppressed alignment and delayed bridge formation. Time-resolved analysis indicates that MXenes assembling is governed by the concurrence of dielectrophoretic forces and PEG-mediated resistance. These findings highlight the interplay influence of electric field parameters, solvent types and matrix properties.

1. Introduction

MXenes, as of their outstanding properties, are paving the way towards development of polymer-based composites provided with excellent electrical, mechanical and optical properties [1–4]. This class of two-dimensional (2D) transition metal carbides, nitrides, or carbonitrides [5] is described by the general formula $M_{n+1}X_nT_x$, where M is an early transition metal (e.g., Ti, V, Nb, Mo), X is carbon and/or nitrogen, T_x denotes the surface functional groups (e.g., -O, -OH, -F), while n ranges from 1 to 4 [6–8]. MXenes are provided with high electrical conductivity, strong electrostatic tunability, high aspect ratios, hydrophilicity, and reasonable colloidal stability in polar solvents as of their surface terminations [9,10].

Incorporation of MXenes can be done directly, according to classical experimental protocols, or, for tailoring the properties of the resulting polymer composites, external controlling means can be employed for their alignment in specific direction with the aim of enhancing

mechanical, electrical, and thermal properties [11].

Thus, aligned MXene-polymer composites have been broadly investigated by utilizing various techniques such as shear-induced alignment [12,13], unidirectional floating film transfer [14], hot-pressing [15,16], vacuum-assisted filtration [17,18] and electric field assisted [19,20]. The electric field assisted alignment is one of the most promising techniques to align MXene flakes (vertically [20–22] or horizontally [24]) as of its simplicity.

Aligning MXene flakes within composites matrices offers substantial performance advantages across a wide range of technological fields [23]. Thus, controlled assembling impacts the electrical conductivity and charge transport (forming continuous conductive pathways) [9], electromagnetic interference shielding and microwave adsorption (controlled layer stacking, interfacial polarization) [9,24,25], mechanical strength and structural integrity (effective load transfer and reinforcement provided by high-aspect ratio flakes embedded in polymer or matrix composites) [26], as well as, thermal management or

* Corresponding author at: "Ion Ionescu de la Brad" Iasi University of Life Sciences, 3 Mihail Sadoveanu Alley, Iasi 700490, Romania.

E-mail address: tudor-alexandru.filip@academic.tuiasi.ro (T.-A. Filip).

thermal-functional behavior (facilitate heat dissipation or controlled thermal flow across the composite) [27,28].

Thus, understanding electric field-induced alignment process and interface engineering is crucial for tailoring MXene-polymer composites according to specific needs. Based on the intended characteristics of the composite MXene concentration, nature of the polymer matrix, and their related ratio should be tuned. Consequently, solvent selection is crucial as it impacts the solubility of both MXene and the polymer [29]. Therefore, for the development of polymer composites engineered via electric field aligned MXenes, various polymer-solvent systems should be envisaged. Under appropriate conditions, even if MXenes can align reasonably within solvents, their orientation may be limited to different degrees as a polymers' higher viscosity is decreasing the orientation velocity [30]. In addition, recent work highlighted that prolonged or repeated contact with specific solvents can deteriorate the mechanical and electrical properties of MXenes, suggesting that solvent-MXenes interactions may induce structural defects or partial degradation [31]. Moreover, in our previous work, we demonstrated that dielectrophoresis (DEP) can be effectively employed to directly assemble pristine MXene flakes, dispersed in water-based dimethyl sulfoxide (DMSO) solution, into ordered and electrically continuous structures bridging the gaps of screen-printed interdigitated microelectrodes (IDEs) [32]. This approach enabled controlled alignment and spatial positioning of the flakes within electrodes' gaps, promoting formation of well-defined conductive pathways with proven functional VOC sensing capabilities. The resulting architectures highlighted also the capability of DEP to guide MXenes tailoring at the microscale, offering a robust technique for integrating solution-processed 2D materials into printed electronic platforms. The performed experiments allowed us to identify optimal frequency ranges which are enforcing the DEP force while minimizing parasitic electrokinetic phenomena, such as AC electro-osmosis (as of electric double layer) and electrothermal (as of Joule heating) effects, which can disrupt the assembly process. Building upon this foundational work which established the electrical continuity and functional VOC sensing of DEP-assembled conductive MXene bridges, the present study shifts the focus toward a structural optimization framework of future MXene composites.

Herein, the influence of different polar solvents and polymeric solutions on MXenes alignment process is investigated and discussed. The most important intrinsic (solvent electrical properties) and extrinsic (dielectrophoretic parameters) factors were varied to achieve optimal (parallel to the electric field direction) alignment of MXenes. Moreover, suitable signal frequencies for avoiding parasitic phenomena (AC electro-osmosis - ACEO, electrothermal effect - ETE) occurring simultaneously with DEP were experimentally identified. Collectively, these findings provide a comprehensive framework for controlling MXenes alignment under AC fields and offer practical guidelines for integrating solution-processed 2D materials into microelectronic and sensing platforms with enhanced structural and functional precision.

2. Materials and methods

2.1. Materials

$\text{Ti}_3\text{C}_2\text{T}_x$ multilayer MXenes were synthesized from commercially available titanium aluminum carbide (Ti_3AlC_2) MAX phase (312, $\geq 99.99\%$ purity and $\leq 100\ \mu\text{m}$ particle size (Sigma-Aldrich, Steinheim, Germany), using LiF-HCl etching according to a procedure adopted from the literature [33]. Hydrochloric acid (HCl) 37%, hydrofluoric acid (HF) 48%, lithium chloride (LiCl) 99%, ethylene glycol (EG), ethanol (EtOH), dimethylsulfoxide (DMSO), isopropanol (IPA), acetonitrile (ACN), propylene glycol (PG), dichloromethane (DCM) and polyethylene glycol (PEG 2000) were purchased from Sigma-Aldrich (Steinheim, Germany) and used as received without further purification.

2.2. Synthesis of multilayered $\text{Ti}_3\text{C}_2\text{T}_x$ MXenes and delamination

The selective etching of the Ti_3AlC_2 MAX phase was carried out on 1 g of material using a mixture of deionized water (DIW), 12 M HCl, and 28.4 M HF in a volumetric ratio of 9:18:3 (mL). The reaction was carried out at 35°C on a magnetic hotplate for 24 h, in an HDPE reaction vessel equipped with a condensation column to safely release the gases generated during the etching process. The reaction mixture was transferred into Eppendorf tubes and subjected to repeated centrifugation at 3300 rpm, until a neutral pH was achieved (approximately four DI water washing cycles) [33].

The resulting sediment was subsequently subjected to the delamination protocol, using 1 g of LiCl and 50 mL of DIW, and was stirred for 1 h at 65°C as outlined in earlier reports [32,34]. The processing of the intercalated suspension consisted of repeated centrifugation at 3300 rpm, during which the clear supernatant was decanted, and the remaining sediment was redispersed. After the first wash, the solution appeared clear, gradually turning darker with each subsequent washing cycle. To obtain few-layer MXene, the sediment was redispersed in DIW in the final stage and centrifuged at 2400 rpm. The resulting black dispersion was collected and subsequently subjected to lyophilization to obtain solid $\text{Ti}_3\text{C}_2\text{T}_x$ MXene. The etching, delamination, and single-to-few-layer MXene preparation procedures were monitored by Verios G4 UC Scanning Electron Microscope (Thermo Scientific, Czech Republic). The SEM micrographs presented in Fig. 1 provide clear morphological evidence confirming the successful progression of each step involved in the etching and delamination of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene from the Ti_3AlC_2 MAX precursor. In Fig. 1a, the pristine Ti_3AlC_2 MAX phase exhibits the characteristic well-defined, densely packed lamellar structure, with smooth, tightly stacked layers indicative of the intact Al-containing MAX precursor (Figure S1a). Following selective etching, one can notice the formation of multilayered $\text{Ti}_3\text{C}_2\text{T}_x$ MXene, (Fig. 1b) where the removal of Al layers results in a visibly expanded, accordion-like morphology (Figure S1b) [5]. This structural delamination is a well-established signature of successful HF/HCl etching, confirming the effective conversion of Ti_3AlC_2 into $\text{Ti}_3\text{C}_2\text{T}_x$. Further processing through LiCl-assisted intercalation and mechanical delamination yields the morphology shown in Fig. 1c, where thin, partially separated or fully isolated flakes are clearly distinguishable. The flakes exhibit irregular edges and significantly reduced thickness, consistent with the formation of single- to few-layer MXene sheets.

2.3. The preparation of the MXene dispersions in different solvents or PEG solutions

Colloidal dispersions containing 0.25 wt% single-to-few-layer MXenes were prepared by dispersing appropriate amounts of lyophilized MXene powder in various solvents, followed by vortex mixing for 30 min prior DEP manipulations. Several solvents were selected for the preparation of MXene colloidal dispersions, as presented in Table S1. The solvents listed in Table S1 exhibit substantial variations in polarity, dielectric constant, and electrical conductivity, parameters that strongly influence MXene dispersion behaviour, as well as flake stabilization, and interfacial interactions. The electrical conductivity of the solvents was measured using a solution Conductivity/TDS (total dissolved solids) meter (Model 4520, Jenway). DIW shows the highest polarity and dielectric constant of all solvents, reflecting its strong ability to stabilize charged MXene surfaces through electrostatic interactions. Its relatively moderate conductivity supports the formation of stable colloidal dispersions, as commonly reported for aqueous MXene systems [35,36].

Ethylene glycol (EG) and propylene glycol (PG) present lower polarity and dielectric constants, compared to DIW, which remain within a range that allows favorable interactions with surface terminations. Their low electrical conductivities indicate reduced ionic mobility, which may affect the screening of surface charges and the resulting colloidal stability [37]. DMSO displays a combination of moderate polarity and a

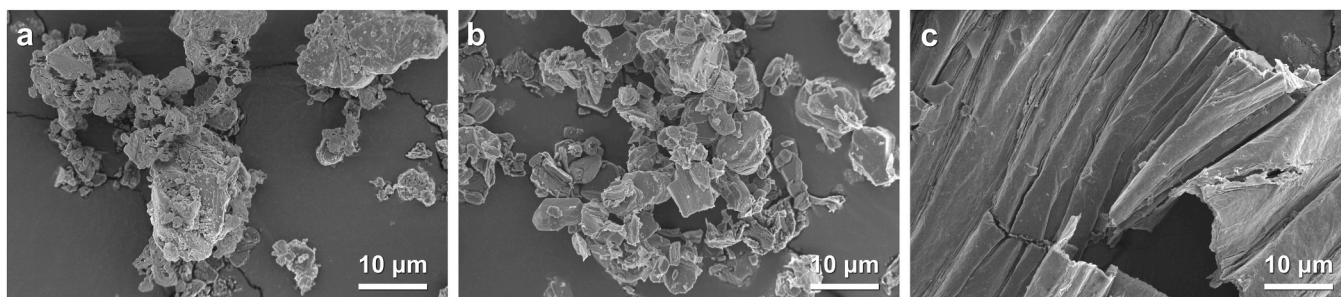


Fig. 1. SEM images of Ti_3AlC_2 MAX phase (a), $\text{Ti}_3\text{C}_2\text{T}_x$ multilayered MXenes (b) and few-layers $\text{Ti}_3\text{C}_2\text{T}_x$ MXene (c).

high dielectric constant, enabling strong dipolar interactions with MXene flakes. Its intermediate conductivity suggests a balanced environment for both electrostatic and dipole-driven stabilization mechanisms [38]. Ethanol (EtOH) and isopropanol (IPA) possess lower dielectric constants and polarities, characteristic of less polar protic solvents. Interestingly, IPA shows a higher conductivity in comparison to EtOH, and limited electrostatic stabilization, even though its overall polarity remains significantly lower than that of water [39]. Acetonitrile (ACN) combines low-to-moderate polarity with a relatively high dielectric constant for an aprotic solvent. This unique combination may support specific dipolar interactions with MXene surfaces, though its modest conductivity may limit electrostatic contributions to stability [38]. Dichloromethane (DCM) exhibits the lowest polarity, dielectric constant, and conductivity. These characteristics make it the least effective solvent for stabilizing MXene dispersions, as weak dipolar interactions and minimal charge screening are expected to lead to rapid sedimentation or aggregation [40].

Besides the dispersions prepared in various pure solvents, two additional samples were developed by dispersing MXene, in the same conditions (0.25 wt%), in PEG 2000 aqueous solutions (DIW) with polymer concentrations of 10 wt% and 15 wt%, respectively, followed by vortex mixing for 30 min prior DEP manipulations. Viscosity measurements of PEG 2000 ($c = 10\%$ w/v, $c = 15\%$ w/v) were carried out in double distilled water at 25°C ($+ 0.01^\circ\text{C}$), on Schott viscometer AVS 350 (Geräte GmbH, Germany), with an Ubbelohde suspended-level viscometer. The kinetic energy corrections were found to be negligible. The flow volume of the viscometer was above 5 mL, making drainage errors unimportant. Flow times were obtained with an accuracy of $+ 0.035\%$, for different measurements.

2.4. Interdigitated electrodes fabrication

A carbon-based conductive ink (solid content: 21.4%, density: 1.09 g/cm^3 , viscosity at 200°C : 10,250 cPs, Loctite) was deposited on top on flexible polyethylene terephthalate (PET, Melinex) substrate with a thickness of 125 μm . The ink was screen-printed via polymeric mesh (100 T) using a medium-hardness squeegee (durometer: 70) to ensure consistent ink transfer and reasonable pattern resolution, with printing parameters carefully optimized to minimize edge bleeding and ensure uniform deposition across the substrate. Following printing, the electrodes underwent thermal treatment to cure the ink and enhance its electrical and mechanical properties. The curing protocol was conducted at 120°C according to ink's rheological characteristics and thermal tolerance of the PET substrate. This process facilitated solvent evaporation and binder crosslinking, resulting in robust, adherent, and conductive electrode structures without compromising substrate integrity.

2.5. Dielectrophoretic application

MXenes alignment between electrode micro-gaps was realized based on a non-uniform electric field generated using a waveform generator

(Keysight 33521 A). The electrodes were connected to the function generator through MicruX drop cell connector (MicruX Technologies, Spain). In all the experiments, the same amount of MXenes sample (1.5 μL) dropped over the active surface of the electrodes. Finally, the drop was covered with borosilicate glass slides using spacers to prevent solvent evaporation. The displacement of MXene flakes within dispersions under non-uniform AC electric field was monitored with a digital microscope (Dino-Lite Edge Digital Microscope AF4915ZTL). To evaluate the efficiency of the dielectrophoretic process, a comprehensive directional analysis was conducted on the steady-state microscopic images for all eight solvent systems using the Directionality plugin (Fourier components method) in ImageJ/Fiji [41,42]. This computational approach allowed the mathematical extraction of the angular distribution profiles and the subsequent calculation of the 2D orientation order parameters ($S = 2 \cdot \langle \cos^2 \Delta\theta \rangle - 1$).

3. Results and discussion

3.1. Effect of the DEP parameters on the MXenes alignment

The alignment of MXenes via DEP depends critically on the amplitude, frequency, and application time of the signal. According to AC electrokinetic theory, an electric field produces extra electrokinetic phenomena beyond DEP, hence the direction of the MXenes motion depends on the signal frequency [43]. These unwanted phenomena may dominate the MXenes behavior at certain frequencies [32]. To assess the status of MXene flakes alignment within polar protic solvents (DIW and EtOH) under the action of the electric gradient at various frequencies, we propose an analysis of the microscopic images (Fig. 2) obtained while applying 20 V peak-to-peak (Vpp) for 2 min. At 100 Hz MXenes were not affected only by pDEP, but rather by its simultaneous action in corroboration with ACEO and ETE, especially in the case of particles dispersed in EtOH. In this case, MXenes are not totally assembling as continuous bridges across finger pairs as the desired pDEP forces are weak and surpassed by the parasitic phenomena. Thus, at this frequency, only part of MXene flakes is free to form small chains along electrode edges as of particle–particle interaction.

At 10 kHz, one can notice the fact that ETE is disappearing while the positive DEP is overcoming the electro-osmotic effect. In fact, DEP and ACEO often compete simultaneously for controlling particle movement, and their relative dominance is frequency dependent. At low frequencies, ACEO is the most effective and dominant in low-conductivity solutions (EtOH) as the low ion density results in a thicker electrical double layer (EDL), which interacts effectively with the electric field to generate flow. At high frequencies (typically above 100 kHz - 1 MHz), ACEO is less significant as the EDL cannot assemble or respond fast enough to the rapidly alternating field. In this regime, DEP becomes the predominant force and particle motion can be controlled mainly by the electric field gradient, without significant interference with ACEO [44]. MXene flakes align when a high frequency electric field is attained as of the fact that the solvent and counterion cluster at the colloidal interface allow formation of imaginary dipole moments [20]. As a result, torque is

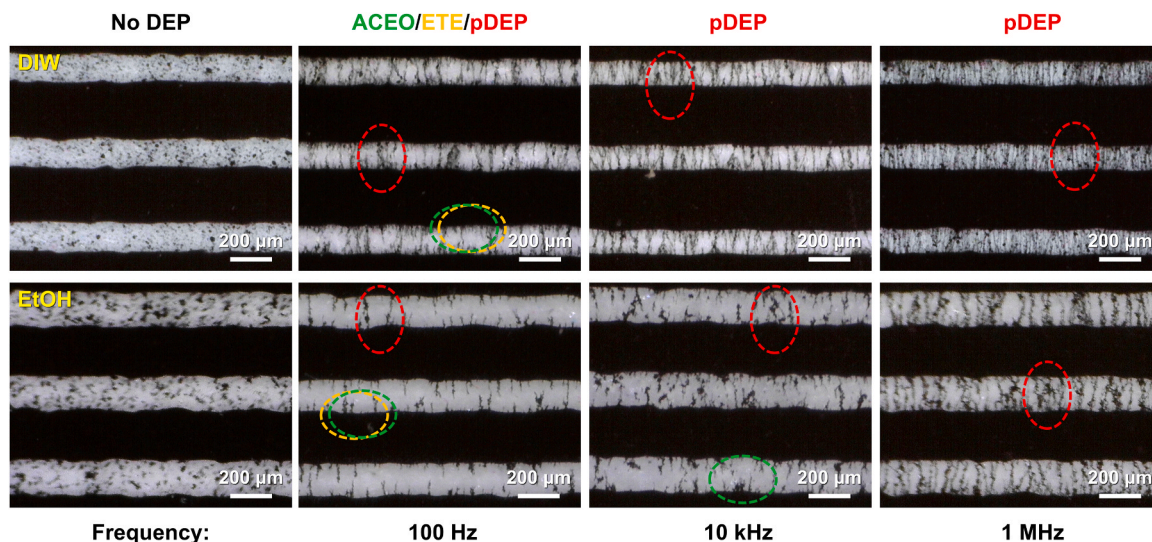


Fig. 2. Microscopic images of MXene flake distributions at the level of IDEs within DIW and ethanol solvents, before and after 2 min of DEP action at different frequencies.

imparted to the particle to align parallel to the electric field, facilitating, thus, a neat and uniform arrangement of MXene nanobundles along the electric field direction.

Fig. 3 depicts time-dependence formation of MXenes bridges at various signal magnitudes (i.e., 10 Vpp, 15 Vpp, 20 Vpp) at 1 MHz. After applying an electric field for 20 s, MXene flakes oriented to form chain structures along the direction of the electric field, even at lower voltage. As both the signal amplitude and the duration of the applied field were increased, the lateral interconnections between individual nanochains became progressively reinforced, leading to structures with larger radial dimensions. This morphological evaluation suggests that stronger DEP forces promote enhanced particle mobility and more frequent inter-chain encounters, facilitating formation of secondary junctions between adjacent assemblies. Case the electric field is applied for 120 s, the local concentration of MXene flakes within the active region is

substantially increased, enabling chains to merge into thicker/wider bridges. This effect reflects a transition from initial, loosely connected chain segments to more consolidated conductive pathways, driven by the combined effects of sustained particle polarization, field-induced dipole interactions, and reduced inter-chain spacing. Our previous observations highlight the dynamic nature of DEP-mediated assembly and underscore the importance of both field strength and exposure time in dictating the structural robustness and continuity of MXenes architecture.

3.2. Effect of solvent selection on the MXenes alignment

According to previously described results, the medium properties directly affect the DEP force, ETE force (due to Joule heating), and ACEO flow (due to the electric double layer).

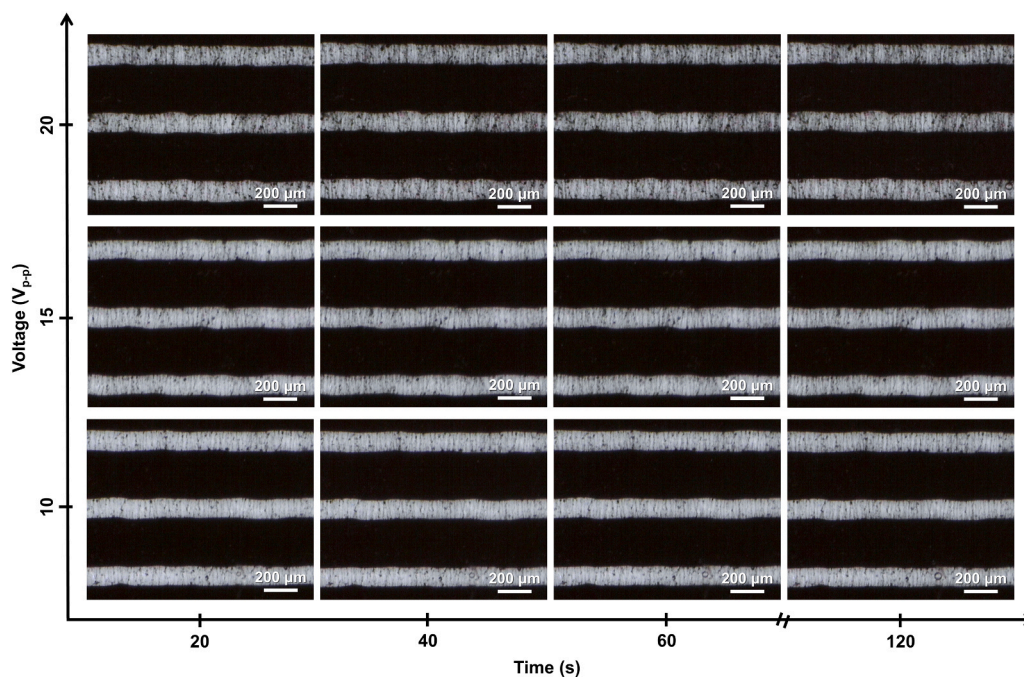


Fig. 3. Time-dependence formation of MXene bridges in DIW across electrode gaps at different signal magnitudes (10, 15 and 20 Vpp) and at fixed frequency of 1 MHz.

Although the literature simulations proposes the suitable DEP medium based on its electrical and thermal properties, it cannot predict the dispersity of particles in different mediums or stability of solvent for a long and uniform alignment process [45,46].

For example, part of the solvents, as Methylene Chloride, Acetone and Ethyl Acetate are not suitable for long-duration DEP alignment due to their high evaporation rates. The hydrophilic nature of MXene flakes, as of their terminal functional groups, presents a significant challenge for achieving stable colloidal dispersion in many organic solvents, particularly in non-polar ones [47,48]. This inconvenient leads to pronounced aggregation and re-stacking of the flakes, which compromises the deaggregation of 2D material within the solvent and represents a critical barrier to their application, especially since processing in organic media is often sought to mitigate the oxidative degradation observed in aqueous environments [48,49]. Thus, experimental strategies envisaged direct solvent exchange into polar organic solvents (e.g. like dimethylformamide (DMF) [49] to covalent surface modification with moieties such as alkoxides), which enabled dispersion in non-polar solvents as toluene by preventing flake re-stacking [50]. The development of universal ligands further extends this capability, allowing for the functionalization and stable dispersion of various MXenes types across a broad spectrum of organic solvents, thereby enhancing their processability and stability [47].

As depicted within Fig. 4, the alignment of MXene flakes was not observed in DCM and PG at 20 Vpp and 1 MHz, whereas different degrees of orientation were observed in other solvents. The DIW served as the optimal solvent for aligning MXene particles which assembled as chains along the electric field direction, resulting in the most regular and ordered arrangement. Results suggest that MXenes aligned in solvents with high relative dielectric constant, as DIW, DMSO, EtOH, ACN, IPA and EG and not in solvents with low relative dielectric constant, as DCM. The explanations stands in the fact that particles align under DEP in high-dielectric constant solvents as these media are reducing the electrostatic interactions allowing the applied electric field to polarize the particles and exert a proper force on target objects. In low-dielectric constant solvents, the charges are not electrostatically shielded, leading to strong ion-pairing and reduced particle polarization, fact which is weakening the DEP effect and prevents alignment [51]. However, some counterexamples have shown that MXenes can align in IPA, but less in EG and PG, although the former solvent exhibits a higher relative dielectric constant than other ones. This indicates that other factors, such as polarizability or conductivity of the solvent, affect the orientation of MXene flakes. When an electric field is applied, a solvent molecule with high polarizability's center is more likely to shift, creating polarizable entities [52] that diminishes the initial electric field strength by inducing an electric field which is opposing the external electric field [53]. For example, the polarizability of EG (7.9) is higher than that of

IPA (5.46), thus, it facilitates the formation of polarizable charges and weakens the original electric field strength, preventing the electrophoretic alignment of MXenes.

To provide an objective mathematical validation of these solvent-dependent behavioral trends, a rigorous quantitative directional analysis was done on the steady-state micrographs. The resulting angular distribution profiles and their corresponding orientation order parameters (S) are systematically compiled in Fig. 5 and Table S2. This data fully corroborates the qualitative observations, successfully partitioning the investigated processing media into three distinct groups based on their alignment efficiency (Table S2).

Group I encompasses the high-efficiency alignment solvents, strictly led by DIW ($S = 0.87$), followed by ACN ($S = 0.68$) and EtOH ($S = 0.64$). The orientation profiles for this group display exceptionally sharp and narrow Gaussian peaks centered symmetrically at the 0° deviation axis (i.e. parallel to the electric field direction). This mathematical signature proves that the intense positive dielectrophoretic torque completely overcomes both Brownian motion and local thermal fluctuations, ensuring a near-perfect rectification of the 2D flakes parallel to the vertical electric field lines.

Conversely, moderate, viscosity-limited alignment behavior (Group II) is quantitatively identified for IPA ($S = 0.59$), EG ($S = 0.41$), and DMSO ($S = 0.40$). For these solvents, the angular spectra degrade into considerably broadened Gaussian envelopes, with the alignment dispersion spreading significantly between -30° and $+30^\circ$. In the case of EG and DMSO, this structural deviation is heavily governed by the high intrinsic viscosity of the bulk medium (≈ 21 mPa·s for EG and ≈ 2.0 mPa·s for DMSO). The elevated hydrodynamic drag exerts a strong mechanical resistance against the rotational torque induced by the AC field, effectively trapping a sub-fraction of the MXene flakes in misaligned states before complete equilibrium is attained.

Finally, Group III marks the complete breakdown of the dielectrophoretic assembly, as mathematically certified by PG ($S = 0.29$) and DCM ($S \approx 0$). The orientation profile for DCM remains entirely flat and isotropic across the whole angular spectrum -90° to $+90^\circ$. This absolute flatline serves as quantitative evidence that in low-permittivity media, particle polarization is fully suppressed by ion-pairing, reducing the active DEP force to negligible levels.

3.3. MXenes alignment within polymeric solutions

Building on the insights of MXene alignment in simple solvents, within this section we examined the dielectrophoretic alignment of MXenes within polymeric matrices, as a potential mean for tailoring composites' performance [53]. The above-presented results outlined that MXenes exhibited pronounced colloidal stability and directional alignment in DIW which is a convenient solvent for polymers as PEG,

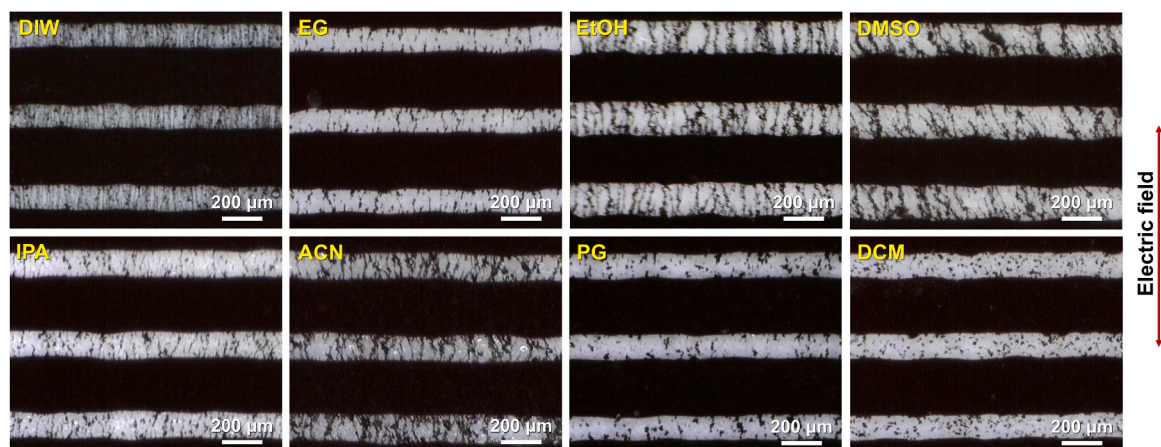


Fig. 4. Effect of the solvent type on the alignment of MXene flakes under an electric field (signal magnitude: 20 Vpp, frequency: 1 MHz, action time: 2 min).

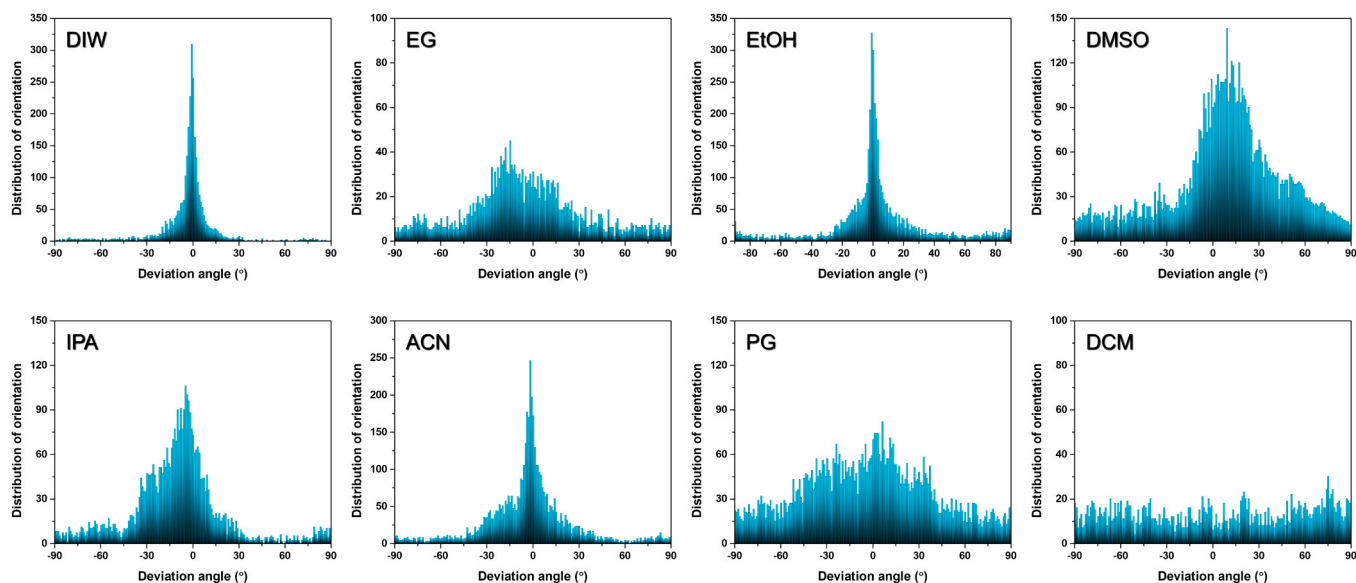


Fig. 5. Quantitative directional analysis of MXene alignment under the applied AC electric field (signal magnitude: 20 Vpp, frequency: 1 MHz, action time: 2 min) across different solvents.

polyvinyl alcohol (PVA) and polyacrylamide (PAM). Among them, PEG 2000 exhibits characteristic polymer–solvent interactions that strongly influence their physicochemical behavior. PEG 2000 is highly water-soluble as of extensive hydrogen bonding between its ether oxygen atoms and water molecules, resulting in clear and homogeneous solutions across a wide concentration range [54]. As PEG concentration increases, the viscosity of the solution rises noticeably as of enhanced chain entanglement and reduced mobility of both polymer coils and surrounding water molecules. PEG solutions also show modest increases in respect to density and refractive index relative to pure water, reflecting the contribution of the polymer to the overall mass and optical properties of the medium [55]. Thermodynamically, PEG 2000 solutions display near-ideal mixing at low concentrations, while higher concentrations can lead to slight deviations associated with polymer-polymer interactions. Overall, the hydrophilic and flexible nature of PEG 2000 imparts predictable and smoothly varying solution properties, making it widely used for colloidal stabilization, dispersion preparation, and viscosity tuning in aqueous systems [56]. In the context of thermal energy storage and battery thermal management, integrating PEG into structured, MXene-modified dual networks or anisotropic composite aerogels has recently emerged as an effective strategy to secure shape-stability

and high latent enthalpy [57]. Compared to these multi-component scaffolding and freeze-casting approaches, the in situ electric-field-driven assembly strategy presented in this work offers a distinct alternative by actively rectifying the MXene flakes into continuous, highly parallel conductive pathways without reducing the volumetric packing density of the polymer matrix, thereby potentially maximizing the overall thermal energy storage density.

In our experiment, PEG with different concentrations (10 wt% and 15 wt%) was selected to explore the DEP alignment of the MXenes in the polymer solution at 20 Vpp and 1 MHz. Microscopic observations were conducted at defined time points (20 s, 40 s, 60 s, and 120 s), enabling a temporal assessment of the morphology and DEP-driven organization (Fig. 6). To quantitatively evaluate the effect of PEG concentration on MXene mobility, the viscosity of 10 and 15% (w/v) PEG 2000 solutions was determined at 25 °C. The 10% PEG solution exhibited a flow time of 201.25 s and a density of $1.01327 \text{ g}\cdot\text{cm}^{-3}$, corresponding to a kinematic viscosity of $2.0125 \text{ mm}^2\cdot\text{s}^{-1}$ and a dynamic viscosity of 2.0392 mPa.s. Increasing the PEG concentration to 15% resulted in a flow time of 276.33 s and a density of $1.02219 \text{ g}\cdot\text{cm}^{-3}$, corresponding to a kinematic viscosity of $2.7633 \text{ mm}^2\cdot\text{s}^{-1}$ and a dynamic viscosity of 2.8246 mPa.s. Therefore, the increase in PEG concentration from 10% to 15% resulted

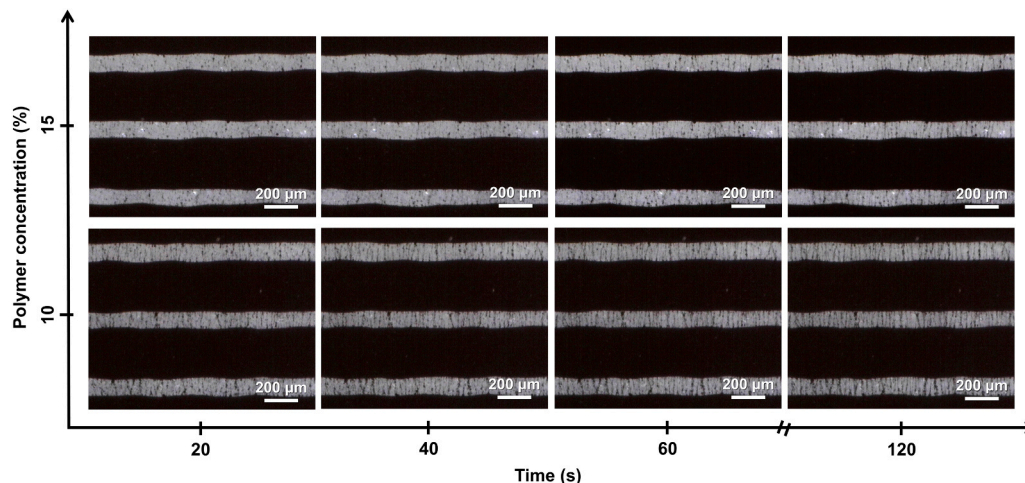


Fig. 6. Time-dependence alignment of MXenes in 10 and 15% PEG solution under an electric field (signal magnitude: 20 Vpp, frequency: 1 MHz).

in an approximately 38.5% increase of dynamic viscosity. These quantitative measurements firmly support the hypothesis that the higher polymer concentration imposes a greater hydrodynamic resistance on the MXene flakes during electric-field-induced transport. As shown in Fig. 6, the electric field applied for 20 s induced the formation of well-defined MXene nanobundles within the 10% PEG matrix ($\eta = 2.0392$ mPa·s). This rapid structural organization suggests that the lower polymer concentration provides sufficient particle mobility and electrostatic responsiveness for the MXene flakes to overcome viscous drag and quickly assemble into ordered, continuous chains. In contrast, at the higher PEG concentration (15%, $\eta = 2.8246$ mPa·s), such nanobundle formation is completely unobservable at the early 20 s stage. This latent response indicates that the 38.5% increase in dynamic viscosity and the associated polymer chain density significantly hinder the initial MXene reorientation, fully suppressing DEP-induced structural ordering within the same time window. As the polymer matrices possess a higher viscosity than pure solvents, the alignment velocity drops as a function of concentration due to the enhanced Stokes hydrodynamic resistance opposing both the rotational and translational motion of the 2D flakes. Moreover, the denser polymer network at 15% PEG physically restricts interflake mobility, dampening the effective electro-orientational torque generated by the applied electric field.

However, as the voltage application time is extended to 40 s and 60 s, respectively, the field-induced assembly of MXenes in the lower-viscosity 10% PEG solution becomes progressively more visible and structurally pronounced. In sharp contrast, extending the application time to 60 s for the MXenes dispersed in the 15% PEG matrix fails to foster visible assembly, with clearly distinguishable MXene bridges and interconnected paths becoming apparent only after a prolonged period of approximately 120 s. This heavily delayed response indicates that the higher polymeric concentration imposes substantial viscous and steric constraints that markedly slow down the alignment kinetics.

The time-dependent observations highlight that the dynamic balance between DEP forces and PEG-mediated solutions influence the progress of MXene assembly, underscoring the critical role of polymer-solvent influence in dictating the kinetics and quality of field-induced chains. Furthermore, the distinct temporal responses observed across PEG concentrations denotes that MXenes alignment is highly sensitive to subtle variations in microstructural environment, revealing an interplay between flake mobility and velocity, and local polymer dynamics that ultimately shapes the morphology of the aligned flakes.

4. Conclusions

MXenes have attracted significant academic interest as functional fillers for polymer composites development as of their exceptional electrical, mechanical, and optical properties as well as their highly tunable surface chemistry, which enables precise control over final composite performance. Despite this potential, technological challenges - particularly particle agglomeration and difficulties in exfoliation or unbundling - continue to limit the development of high-performance MXene-based composites. These issues become even more critical when fabricating anisotropic composites, where controlled particles alignment is required. As solvent properties significantly influence MXenes dispersion and assembly - especially under externally applied electric fields - the herein work presents a set of experimental practices derived from systematic in-lab investigations aimed at identifying the most suitable solvents for MXene-polymer composite fabrication.

Our results highlight that DIW serves as the most effective medium for dielectrophoretic alignment, enabling MXene flakes to assemble into chains oriented along the electric field direction. This confirms that solvent conductivity and polarizability strongly affect MXene orientation behavior. Furthermore, we provide an in-depth analysis on how PEG concentration modulates the dielectrophoretic forces governing MXenes assembly. Our findings demonstrate a clear dependence of MXene chains and nanobundles formation on PEG concentration. Time-

resolved observations also reveal that the evolution of MXene-based structures (bridges) is dictated by a dynamic balance between dielectrophoretic forces and PEG-mediated solution effects.

Collectively, the field-driven assembly framework established herein provides practical guidelines for integrating solution-processed 2D materials into microelectronic and polymer platforms with high structural precision. Looking forward, the precise electro-manipulation of MXene alignment opens promising avenues for the development of anisotropic phase change materials (PCMs) and smart thermal management systems, where the aligned 2D networks can be utilized to optimize directional heat dissipation while leveraging the polymer matrix to suppress mechanical relaxation and leakage during energy storage.

CRediT authorship contribution statement

Ina Turcan: Writing – original draft, Writing – review & editing, Formal analysis, Visualization, Validation, Resources, Project administration, Methodology, Investigation, Funding acquisition, Data curation, Conceptualization. **Tudor-Alexandru Filip:** Writing – review & editing, Writing – original draft, Validation, Resources, Investigation, Formal analysis, Data curation. **Tăchiță Vlad-Bubulac:** Writing – original draft, Validation, Resources, Investigation, Data curation, Conceptualization. **Daniela Rusu:** Writing – review & editing, Validation, Resources, Investigation, Formal analysis, Data curation. **Marius Andrei Olariu:** Writing – review & editing, Writing – original draft, Validation, Supervision, Resources, Methodology, Conceptualization.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Ina Turcan reports financial support was provided by Romanian Government Ministry of Education. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.materresbull.2026.114411](https://doi.org/10.1016/j.materresbull.2026.114411).

Data availability

Data will be made available on request.

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