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(54) Title: MXENE MATERIALS WITH ENHANCED STABILITY

(57) Abstract: Provided is a method, comprising: providing a solution of delaminated MXene material and a first ionic intercalant; and contacting the solution with a second ionic intercalant so as to exchange the first ionic intercalant and the second ionic intercalant so as to give rise to flocculated portions of the MXene material and a supernatant that comprises the first ionic intercalant and the second ionic intercalant. Also provided is a MXene dispersion, comprising a plurality of portions of single-layer and/or few-layer MXene dispersed in a solvent, the solvent comprising a first ionic intercalant and a second ionic intercalant. Also provided is a stable MXene dispersion, comprising a plurality of portions of single-layer and/or few-layer MXene dispersed in a solvent, the solvent comprising an ionic intercalant, the ionic intercalant optionally being present in supersaturated form.



MXENE MATERIALS WITH ENHANCED STABILITY

CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] The present application claims priority to and the benefit of United States patent application no. 63/231,333, “MXene Materials With Enhanced Stability” (filed August 10, 2021), the entirety of which application is incorporated herein by reference for any and all purposes.

GOVERNMENT RIGHTS

[0002] This invention was made with government support by the Fluid Interface Reactions, Structures and Transport (FIRST) Center, an Energy Frontier Research Center (EFRC) funded by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences. The government has certain rights in the invention.

TECHNICAL FIELD

[0003] The present disclosure relates to the field of MXene materials.

BACKGROUND

[0004] Vanadium carbide MXenes, e.g., V_2CT_x , have shown promise for applications ranging from energy storage and sensing, to electronics and optics. In the past decade, however, research involving V_2CT_x has been limited to the material's multilayered form because of the instability of delaminated V_2CT_x in the material's colloidal state. Accordingly, there is a need in the art for stabilized forms of MXenes, including vanadium carbide MXenes.

SUMMARY

[0005] In meeting the described long-felt needs, the present disclosure provides methods, comprising: providing a solution of delaminated MXene material and a first ionic intercalant; and contacting the solution with a second ionic intercalant so as to exchange the first ionic intercalant and the second ionic intercalant so as to give rise to

flocculated portions of the MXene material and a supernatant that comprises the first ionic intercalant and the second ionic intercalant.

[0006] Also provided are MXene dispersions, comprising a plurality of portions of single-layer and/or few-layer MXene dispersed in a solvent, the solvent comprising a first ionic intercalant and a second ionic intercalant.

[0007] Further provided are stable MXene dispersions, comprising a plurality of portions of single-layer and/or few-layer MXene dispersed in a solvent, the solvent comprising an ionic intercalant, the ionic intercalant optionally being present in supersaturated form.

BRIEF DESCRIPTION OF THE DRAWINGS

[0008] In the drawings, which are not necessarily drawn to scale, like numerals may describe similar components in different views. Like numerals having different letter suffixes may represent different instances of similar components. The drawings illustrate generally, by way of example, but not by way of limitation, various aspects discussed in the present document. In the drawings:

[0009] FIGs. 1A-1F. (FIG. 1A) Schematic of MXene synthesis process. (FIG. 1B) XRD patterns of V_2AlC MAX and multilayer V_2CT_x made by HF etching and mixed acid synthesis methods. (FIG. 1C) Optical image of first supernatant (green) after washing. SEM images of the (FIG. 1D) V_2AlC MAX phase, (FIG. 1E) multilayer V_2CT_x from HF etching, and (FIG. 1F) multilayer V_2CT_x from mixed acid etching.

[0010] FIGs. 2A-2E. (FIG. 2A) Delaminated d - V_2CT_x solution (diluted). SEM images of TBA- V_2CT_x flakes from (FIG. 2B) HF etching and (FIG. 2C) HF/HCl etching on anodized aluminum oxide (AAO). (FIG. 2D) DLS measurements and (FIG. 2E) UV-vis measurements for TBA- V_2CT_x (HF and HF/HCl etch) and TMA- V_2CT_x .

[0011] FIGs. 3A-3D. Optical images of vacuum filtered films made from (FIG. 3A) TBA- V_2CT_x (HF etched) and (FIG. 3B) TMA- V_2CT_x (HF/HCl etched). (FIG. 3C) SEM image of cross-section from the TMA- V_2CT_x vacuum filtered film. (FIG. 3D) XRD patterns of TBA- V_2CT_x (HF and HF/HCl) and TMA- V_2CT_x (HF/HCl etch).

[0012] FIGs. 4A-4E. (FIG. 4A) UV-vis spectra of HF etched TBA- V_2CT_x and oxidized V_2CT_x solution. (FIG. 4B) UV-vis spectra of pristine HF/HCl TBA- V_2CT_x collected at 1 h intervals to show oxidation. TMA- V_2CT_x (HF/HCl etched) films

(FIG. 4C) from pristine solution, (FIG. 4D) from 1 month old solution (stored in an argon filled vial), and (FIG. 4E) from pristine solution aged in air for 1 month after drying.

[0013] FIGs. 5A-5D. (FIG. 5A) Schematic of the ion-exchange procedure. (FIG. 5B) $d\text{-V}_2\text{CT}_x$ solution flocculated using LiCl. (FIG. 5C) XRD patterns of TBA- V_2CT_x , TMA- V_2CT_x , and Li- V_2CT_x made from both samples. These samples were all vacuum-filtered films from the mixed acid synthesis (the TMA and TBA- V_2CT_x patterns are taken from FIG. 3D). (FIG. 5D) Conductivity measurements from vacuum filtered V_2CT_x films with different interlayer ions (TBA^+ , TMA^+ , Li^+). Inset image of vacuum filtered Li- V_2CT_x (TBA) shown. from vacuum filtered V_2CT_x films with different interlayer ions (TBA^+ , TMA^+ , Li^+).

[0014] FIGs. 6A-6D. UV-vis spectra of (FIG. 6Aa) HF etched V_2CT_x and HF/HCl etched V_2CT_x kept flocculated over different periods of time. (FIG. 6B) Concentrated and (FIG. 6C) dilute Li- V_2CT_x solution redispersed after 147 days. (FIG. 6D) Vacuum filtered Li- V_2CT_x film made from 147 day old solution.

[0015] FIGs. 7A-7E. Digital photographs of (FIG. 7A) a large volume of TMA- V_2CT_x solution, (FIG. 7B) a flocculated Li- V_2CT_x solution, (FIG. 7C) a Li- V_2CT_x aerogel, (FIG. 7D) Li- V_2CT_x /PVA hydrogels of increasing MXene concentration (logo credited to Drexel University, College of Engineering), and (FIG. 7E) a Li- V_2CT_x solution spray coated on glass with increasing thickness.

[0016] FIG. 8. Map of etching and delamination procedures performed to arrive at mixed acid etch used in this main text.

[0017] FIGs. 9A-9E. Optical Images of: (FIG. 9A) ice bath setup used for etching V_2AlC , (FIG. 9B) V_2AlC MAX phase, (FIG. 9C) $m\text{-V}_2\text{CT}_x$ synthesized from HF etching. Pressed pellets of multilayer V_2CT_x from (FIG. 9D) HF etching and (FIG. 9E) mixed acid (HF/HCl) etching.

[0018] FIGs. 10A-10E. (FIG. 10A) XRD patterns of different V_2AlC MAX phase powders (the “Conventional MAX” pattern is taken from the main text, panel e of FIG. 1); (FIG. 10B) UV-Vis measurements showing the stability of Li- V_2CT_x (HF/HCl etch, TBA^+ delaminated) from different MAX phase precursors (the “Conventional MAX” spectra was taken from FIG. 5B); SEM images of (FIG. 10C) MAX phase synthesized from traditional conditions outlined in the main text; (FIG. 10D) MAX phase obtained from Carbon Ukraine, Ltd. (FIG. 10E) MAX phase synthesized using an excess (1.6 x

more than traditional synthesis) of aluminum during synthesis. This concept is based on prior experiments with $\text{Ti}_3\text{C}_2\text{T}_x$.¹

[0019] FIG. 11. UV-Vis Spectra of TBA- V_2CT_x etched using the HF method. The sample was kept in the spectrophotometer and spectra were collected at 1-hour intervals.

[0020] FIG. 12. Zeta potential measurements for TMA- and TBA- V_2CT_x , synthesized using the mixed acid etch. These graphs are averages of 5 collected intensity distributions.

[0021] FIGs. 13A-13E. Thermogravimetric analysis in an Ar atmosphere of (FIG. 13A) $m\text{l-V}_2\text{CT}_x$ powder, $d\text{-V}_2\text{CT}_x$ films synthesized from (FIG. 13B) TBAOH, (FIG. 13C) TMAOH, and $\text{Li-V}_2\text{CT}_x$ films synthesized from (FIG. 13D) TBAOH and (FIG. 13E) TMAOH.

DETAILED DESCRIPTION OF ILLUSTRATIVE EMBODIMENTS

[0022] The present disclosure may be understood more readily by reference to the following detailed description of desired embodiments and the examples included therein.

[0023] Unless otherwise defined, all technical and scientific terms used herein have the same meaning as commonly understood by one of ordinary skill in the art. In case of conflict, the present document, including definitions, will control. Preferred methods and materials are described below, although methods and materials similar or equivalent to those described herein can be used in practice or testing. All publications, patent applications, patents and other references mentioned herein are incorporated by reference in their entirety. The materials, methods, and examples disclosed herein are illustrative only and not intended to be limiting.

[0024] The singular forms “a,” “an,” and “the” include plural referents unless the context clearly dictates otherwise.

[0025] As used in the specification and in the claims, the term “comprising” may include the embodiments “consisting of” and “consisting essentially of.” The terms “comprise(s),” “include(s),” “having,” “has,” “can,” “contain(s),” and variants thereof, as used herein, are intended to be open-ended transitional phrases, terms, or words that require the presence of the named ingredients/steps and permit the presence of other ingredients/steps. However, such description should be construed as also describing

compositions or processes as "consisting of" and "consisting essentially of" the enumerated ingredients/steps, which allows the presence of only the named ingredients/steps, along with any impurities that might result therefrom, and excludes other ingredients/steps.

[0026] As used herein, the terms "about" and "at or about" mean that the amount or value in question can be the value designated some other value approximately or about the same. It is generally understood, as used herein, that it is the nominal value indicated $\pm 10\%$ variation unless otherwise indicated or inferred. The term is intended to convey that similar values promote equivalent results or effects recited in the claims. That is, it is understood that amounts, sizes, formulations, parameters, and other quantities and characteristics are not and need not be exact, but can be approximate and/or larger or smaller, as desired, reflecting tolerances, conversion factors, rounding off, measurement error and the like, and other factors known to those of skill in the art. In general, an amount, size, formulation, parameter or other quantity or characteristic is "about" or "approximate" whether or not expressly stated to be such. It is understood that where "about" is used before a quantitative value, the parameter also includes the specific quantitative value itself, unless specifically stated otherwise.

[0027] Unless indicated to the contrary, the numerical values should be understood to include numerical values which are the same when reduced to the same number of significant figures and numerical values which differ from the stated value by less than the experimental error of conventional measurement technique of the type described in the present application to determine the value.

[0028] All ranges disclosed herein are inclusive of the recited endpoint and independently of the endpoints (e.g., "between 2 grams and 10 grams, and all the intermediate values includes 2 grams, 10 grams, and all intermediate values"). The endpoints of the ranges and any values disclosed herein are not limited to the precise range or value; they are sufficiently imprecise to include values approximating these ranges and/or values. All ranges are combinable.

[0029] As used herein, approximating language may be applied to modify any quantitative representation that may vary without resulting in a change in the basic function to which it is related. Accordingly, a value modified by a term or terms, such as "about" and "substantially," may not be limited to the precise value specified, in some cases. In at least some instances, the approximating language may correspond to the

precision of an instrument for measuring the value. The modifier “about” should also be considered as disclosing the range defined by the absolute values of the two endpoints. For example, the expression “from about 2 to about 4” also discloses the range “from 2 to 4.” The term “about” may refer to plus or minus 10% of the indicated number. For example, “about 10%” may indicate a range of 9% to 11%, and “about 1” may mean from 0.9-1.1. Other meanings of “about” may be apparent from the context, such as rounding off, so, for example “about 1” may also mean from 0.5 to 1.4. Further, the term “comprising” should be understood as having its open-ended meaning of “including,” but the term also includes the closed meaning of the term “consisting.” For example, a composition that comprises components A and B may be a composition that includes A, B, and other components, but may also be a composition made of A and B only. Any documents cited herein are incorporated by reference in their entireties for any and all purposes.

[0030] Vanadium carbide MXenes, specifically V_2CT_x , have shown promise for applications ranging from energy storage and sensing, to electronics and optics. In the past decade, however, research involving V_2CT_x has been mostly limited to its multilayered form due to instability of delaminated V_2CT_x in its colloidal state.

[0031] Provided here is, inter alia, a mild synthesis condition approach that results in high-quality MXenes (e.g., V_2CT_x) and an ion exchange process coupled with flocculation that increases the shelf life of the MXene (e.g., in aqueous suspension) by about three orders of magnitude, from a few hours to several months. Without being bound to any particular theory or embodiment, we explain the effect of etchant formulation, delamination chemicals, and post-processing on the quality, chemical stability, and optoelectronic properties of the synthesized MXene.

[0032] As an illustration of the disclosed technology, we demonstrate that V_2CT_x produced using ion-exchange to replace tetrabutylammonium or tetramethylammonium ions with lithium cations and flocculation after delamination can not only be stored in suspension for a few months without degradation, but also can be redispersed and processed into films. Those MXene films show distinct improvements in the optical and electronic properties. Their electrical conductivities in dry state can exceed $1,000 \text{ S cm}^{-1}$, a value not previously achievable for V_2CT_x . The improvements in shelf life and properties of V_2CT_x demonstrated in this work allow fundamental studies of properties of this and other MXenes and greatly expand the range of MXenes applications. The described

approach is applicable to other MXenes, e.g., those for which one can use quaternary amines for delamination.

[0033] Since their discovery in 2011,¹ two-dimensional (2D) transition metal carbides and nitrides, MXenes, have shown promising electrical,² mechanical,³ optical,⁴ and electrochemical properties leading to their widespread use in applications such as energy storage,⁵ electromagnetic interference shielding,^{6,7} sensing,^{8,9} electronics,^{10,11} and biomedicine,¹²⁻¹⁴ to name a few examples.

[0034] MXenes have a general formula of $M_{n+1}X_nT_x$, where M represents early transition metals (Ti, V, Nb, etc.), X is carbon and/or nitrogen, $n=1-4$, and T_x represents the surface functional groups (-O, -OH, -F, -Cl) on these materials.¹⁵ The possibility of having different single or double transition metals, in an ordered or solid solution (random) form, on the M site along with C and/or N in the X site, has resulted in the experimental synthesis of more than 30 stoichiometric MXene compositions with many more computationally predicted.¹⁶ However, despite the numerous MXene compositions available, research has primarily focused on $Ti_3C_2T_x$, mainly because of its high chemical stability and the presence of established synthesis guidelines.

[0035] Among other MXenes, the M_2XT_x structure, in particular V_2CT_x , is interesting due to its larger active area per mass (lower number of atomic layers in their structure; 3 atomic-layers for V_2C vs 5 atomic-layers for Ti_3C_2) with a more chemically active transition metal (vanadium) that has multiple oxidation states. Multilayer (ml - V_2CT_x) or delaminated films of V_2CT_x (d - V_2CT_x) have been used in various applications such as batteries,^{17,18} supercapacitors,¹⁹ gas sensors,²⁰ electronics,²¹ optics,²² and biomedicine.¹⁴ Similar to other MXenes, density functional theory calculations (DFT) have suggested that bare and F/OH terminated V_2C should show metallic behavior,²³ however, because of the presence of large organic intercalants (used for delamination) in the interlayer space of V_2CT_x flakes, experimental measurements have shown semiconductor-like electronic behavior and a negative temperature-dependent resistivity change for V_2CT_x multilayer films.²⁴ The unusual electronic properties of d - V_2CT_x have rendered it promising for various optoelectronic applications. For example, transparent conductive electrodes made from spin-coated thin films of V_2CT_x have shown a higher figure of merit (FOM) compared to other M_2XT_x MXenes, such as Ti_2CT_x .²¹

[0036] V_2CT_x is anecdotally known to be the least chemically stable MXene in its delaminated form. Single and few-layer V_2CT_x flakes readily degrade in water

dispersions or when exposed to air. Because of this instability, previous studies have mostly focused on *ml*-V₂CT_x rather than *d*-V₂CT_x.²⁵ In addition, V₂CT_x is commonly delaminated by large organic molecules such as tetramethylammonium hydroxide (TMAOH) or tetrabutylammonium hydroxide (TBAOH). Because complete removal of TBA⁺ or TMA⁺ ions is difficult to achieve, produced V₂CT_x films usually show a large interlayer spacing. This, along with highly defective nature of V₂CT_x flakes^{17,19} as well as its other intrinsic properties, have so far resulted in lower electrical conductivities (140 S cm⁻¹) compared to Ti₃C₂T_x.^{21,26,27}

[0037] To overcome the chemical instability of V₂CT_x (and other less stable MXenes, such as Ti₂CT_x), numerous post processing approaches including the addition of polyanionic salts to hinder edge-driven oxidation, adjustment of the colloidal solution pH with buffers, and antioxidants have been attempted.^{28,29} Although these approaches lead to improved MXene chemical stability in water dispersions, they can negatively affect the electronic and electrochemical properties. Using buffers in the solution, whether acidic or basic, decreases the electrical conductivity of vacuum filtered films. Moreover, the use of alkaline buffers led to faster MXene degradation over time.²⁹ In addition to post-processing treatments, the quality of the MAX phase precursor, and initial etching and delamination conditions are also important factors for the synthesis of higher quality and chemically stable V₂CT_x flakes, which has already been shown for Ti₃C₂T_x.^{27,30} Therefore, to practically use V₂CT_x for research purposes or device applications, after confirming proper stoichiometry and particle size of the V₂AlC MAX phase precursor, first, the etching and delamination conditions should be improved to produce higher quality V₂CT_x flakes. Second, proper protocols should be developed to improve its chemical stability without negatively affecting its properties. The combination of these two improvements will alleviate existing challenges facing the community, enabling more widespread use of V₂CT_x.

[0038] In this disclosure, we discuss the improved etching and delamination protocols for the synthesis of V₂CT_x and show that, through an ion exchange approach couple with flocculation, *d*-V₂CT_x flakes can be stored for several months with no significant degradation occurring even after ~150 days. We use a mixed acid etchant formulation to synthesize *ml*-V₂CT_x, followed by delamination with TBAOH or TMAOH. A LiCl ion-exchange/flocculation process is used as a post-processing technique to stabilize the *d*-V₂CT_x flakes. This approach both improves its chemical stability and nearly

doubles the electrical conductivity of V_2CT_x freestanding films. We attribute these enhancements to the replacement of the TBA^+ or TMA^+ with Li^+ ions, indicated by a significant reduction in the d -spacing of the MXene films, and their increased conductivity above $1,000\text{ S cm}^{-1}$. It should be understood, however, that although the disclosed technology is illustrated by non-limiting application to V_2CT_x , the disclosed technology is applicable to MXenes in general and is not limited to V_2CT_x .

[0039] Discussion of Methods

[0040] Precursor and Etching Conditions

[0041] Research on $Ti_3C_2T_x$ has shown that properties and morphology of the prepared MXene depend on the stoichiometry, quality, and particle size of the MAX phase precursor.¹⁵ For example, it was recently shown that increasing Ti_3AlC_2 MAX phase quality by controlling its stoichiometry leads to less defective $Ti_3C_2T_x$ flakes with enhanced chemical stability.³⁰ Similarly, the successful synthesis of high quality V_2CT_x depends on the stoichiometry and quality of the V_2AlC MAX phase precursor. For the synthesis of V_2AlC , vanadium (99.5%, -325 mesh), aluminum (99.5%, -325 mesh), and graphite (99%, -325 mesh) powders are used.

[0042] First, V, Al, and C precursors are mixed in a 2:1.1:0.9 atomic ratio (e.g., 50 g total per batch). The precursors are ball-milled with 10 mm yttria stabilized zirconia balls (2:1 ball: powder mass ratio) in plastic jars at 60 rpm for 18 h to ensure a homogeneous mixture of the powders. The powder mixture is transferred into alumina crucibles and placed into a high-temperature tube furnace (Carbolite Gero). The furnace should be purged with ultra-high purity Ar gas (200 SCCM) prior to heating for at least 1 hour. Argon should also be continually flown through the furnace throughout the sintering procedure. The furnace is heated to $1550\text{ }^\circ\text{C}$ at a rate of $3\text{ }^\circ\text{C min}^{-1}$, held for 2 h, and then cooled to room temperature at a rate of $3\text{ }^\circ\text{C min}^{-1}$. Afterwards, the sintered compact can be milled using a TiN-coated bit or crushed with a mortar and pestle, then sieved to the desired particle size (usually $< 38\text{ }\mu\text{m}$ (400 mesh)). The sieving process ensures a uniform particle size distribution and similar etching kinetics between the MAX particles. The obtained V_2AlC particles usually contain unreacted elemental powders or intermetallic impurities. They can be removed by acid washing the MAX powders in 9 M HCl for 12 hours. 2 mL of 9 M HCl were used for every gram of V_2AlC . Afterwards, the V_2AlC particles are washed by filtration with deionized water until a pH of >5 - 5.5 is achieved.

[0043] FIG. 1A shows different steps involved for the synthesis of V_2CT_x . Typically, V_2AlC is etched in 48-50% hydrofluoric acid (HF). To address certain challenges associated with using concentrated HF, we developed a milder etchant based on a mixture of HF and hydrochloric acid (HCl) for the synthesis of V_2CT_x , the details of which is provided in the following text.

[0044] Etching can be done in high density polyethylene (HDPE) bottles and per 1 g of V_2AlC , 20 mL of etching solution (either pure HF or HF/HCl as described below) was used. To ensure safety and proper mixing, the etching bottle can be selected so that the etchant does not occupy more than one third of its volume. For example, 1g V_2AlC can be etched in a 60 mL HDPE bottle, but if 2 g V_2AlC powder is required to be etched, the volume of etchant solution will be 40 mL and therefore, a larger HDPE bottle (i.e. 125 mL) should be used. Moreover, it is useful to use an ice bath for the addition of V_2AlC because the highly reactive particles can experience local heating (as the result of exothermic reaction between MAX phase powder and the acidic etchant) which can cause instant oxidation/dissolution. Therefore, after placing the etchant bottle in an ice bath (FIG. 9a), slowly, 1 g of V_2AlC is added to the etchant while stirring the solution at low speed (150 rpm). The addition of powder can take appx. 5 min per gram of MAX phase.

[0045] After all powder is added, one can monitor the reaction for 1-2 min to make sure no severe reaction is occurring (no bubbling should be observed). Then the bottle is loosely capped, and subsequently transferred to an oil bath to start the etching in a controlled temperature condition. Stirring speed can be fixed at 400 rpm for all etching conditions. The proper safety precautions should be followed when working with HF, as recently outlined.³²

[0046] We investigated the effect of etching time of V_2AlC in HF and HF/HCl solutions and determined the minimal time required for most complete conversion of V_2AlC into V_2CT_x without inherent oxidation. The table in FIG. 8 outlines the different etching configurations tested. The premise of a mixed acid etch was based on previous work with $Ti_3C_2T_x$.³³ For the conventional HF etching protocol, 20 mL of 48 wt. % HF is used per 1 g of MAX phase, and the reaction proceeds for 96 hours at 25 °C.¹⁸ For the mixed acid method, 1 g of V_2AlC is added to a mixture of 12 mL of 48 wt. % HF and 8 mL of 12 M HCl, and the reaction proceeds for 72 hours at 50 °C.

[0047] After etching is completed, the etchant mixture is diluted and transferred to 175 mL centrifuge tubes and centrifuged at 3500 rpm (2550 rcf) for 5 minutes. At this

point, the ml - V_2CT_x , along with any remaining MAX phase, will be sedimented at the bottom of the tube and a green supernatant, which is caused by vanadium ions in solution, is obtained (FIG. 1b). It is important to note that the green supernatant observed at this stage is not MXene, but is instead dissolved V from small intermetallics, residual metal, and nanosized MAX phase present in the precursor. Therefore, it should be decanted as waste.

[0048] The multilayer powder was washed with DI water and centrifuged repeatedly until the pH of the supernatant was >5.5 . The washing typically required >1.5 L of deionized water per 1 g etched MAX phase. The ml - V_2CT_x can be immediately delaminated (using the wet powder), dried for storage, or used and processed directly.

[0049] The ml - V_2CT_x powder exhibits a dark brown color compared to the grey MAX phase, which can be seen in FIGs. 9B and 9C respectively. There is no apparent optical difference between the loose ml - V_2CT_x powders etched by HF or mixed acid etching, but when the multilayer powders are pressed into pellets, the mixed acid etch is brighter and more golden in color (FIGs. 9D, 9E). X-ray diffraction (XRD) patterns were collected at each stage of synthesis for both etching methods and are shown in FIG. 1C. For preparation of XRD samples, V_2CT_x was prepared simultaneously using both etching methods, followed by identical drying procedures. The multilayer powders were dried on a vacuum filter, followed by 24 hours in a vacuum desiccator at RT. The powders were subsequently crushed into a fine powder and dried for an additional 48 hours in the vacuum desiccator. 200 mg of each multilayer powder was pressed into pellets at 50 MPa to orient the MXene powders along their basal planes. The shift of the (002) peak, from 13.44° in V_2AlC to 9.22° (mixed acid etch) and 8.98° (HF etch) for ml - V_2CT_x is attributed to the successful removal of Al from V_2AlC and subsequent expansion of the $d_{(002)}$ -spacing from 6.6 Å to 10 Å and 9.6 Å, respectively. The intensity of the MXene (002) peak relative to the MAX phase peaks is greater in the mixed acid etch, which is indicative of a more complete etching of the Al layers with higher yield of V_2CT_x MXene from the mixed acid etch. These XRD characteristics can be directly compared between the samples due to the identical precursor, processing conditions, and sample preparation. The SEM images in FIGs. 1D-1F compare the layered structure of the V_2AlC MAX phase with the obtained ml - V_2CT_x . Both ml - V_2CT_x MXenes show an open and typical “accordion-like” structure (Fig. 1E-1F).

[0050] Delamination and Ion-Exchange Process

[0051] The ml - V_2CT_x powders are delaminated using tetrabutylammonium hydroxide (TBAOH; denoted TBA- V_2CT_x), or tetramethylammonium hydroxide (TMAOH; denoted TMA- V_2CT_x). For best results (higher yield), delamination should be done immediately following etching and using the wet multilayer powder, however, it is also possible to delaminate vacuum-filter dried MXene powders. In the latter case, one can store the dried ml - V_2CT_x powder under inert atmosphere (i.e. inside Ar-filled glovebox), and take it out when the delamination will be done.

[0052] The delamination protocols for TMAOH were as follows: ~ 1 g of freshly etched ml - V_2CT_x powder (in wet state) was added to 20 mL of a 5 wt. % TMAOH solution in water, and the solution was stirred at 400 rpm at room temperature (25 °C) for 6 hours. Alternatively, when dried ml - V_2CT_x is used for delamination, 200 mg powder can be added to 10 mL of a 25 wt. % TMAOH solution in water, and the solution should be stirred at 400 rpm at 35 °C for 6 hours. Different delamination conditions are used because fresh (wet) multilayer MXene has preintercalated water between the layers, giving better ion mobility and intercalation,³⁴ enabling the process to occur more efficiently.

[0053] When MXene multilayer powders are dried, the interlayer spacing and ion mobility between the layers decreases as the result of water deintercalation, leading to sluggish delamination kinetics and lower yield. Therefore, to achieve a higher yield (~ 50 %), one can use the freshly etched wet powders to prepare dispersions of d - V_2CT_x . Similar to TMAOH, the delamination protocols for TBAOH are: ~ 1 g of freshly etched ml - V_2CT_x powder was added to 20 mL of a 5 wt. % TBAOH solution in water, and the solution was stirred at 400 rpm at 25 °C for 6 hours. Alternatively, 200 mg of dried ml - V_2CT_x was added to 10 mL of a 40 wt. % TBAOH solution in water, and the solution was stirred at 400 rpm at 35 °C for 6 hours.

[0054] After stirring in the delaminating agent, the intercalated multilayer powder is transferred to a centrifuge tube filled with DI water. The solution is centrifuged at 3500 rpm (2550 rcf) for 10 minutes and the first supernatant was discarded. The sediment is dispersed in DI water and shaken by hand for 2 minutes before centrifuging at 2500 rpm (1300 rcf) for 10 minutes (higher centrifuge speeds result in lower concentration of d - V_2CT_x supernatant). It is crucial that the shaking process be performed for at least two minutes to ensure that the sediment is uniformly redispersed in fresh DI water. The second supernatant is once again discarded. After this stage, redispersion, hand shaking, and the 2500 rpm (1300 rcf) centrifugation is repeated with the supernatant being collected until

the d -V₂CT_x concentration is low (when supernatant turns to a blue-green color or becomes transparent). This process can use ~1.2 L of water for 1g of MXene.

[0055] In FIG. 2a, the dilute TBA-V₂CT_x solution (20 mL vial) is green-blue, while FIG. 2b,c shows SEM images of the TBA-V₂CT_x flakes obtained from both HF and mixed acid etching methods drop-cast on an anodized aluminum oxide (AAO) membrane. Notably, depending on the type of the intercalant used, flakes with different physical properties are obtained; TMA⁺ molecules are smaller than TBA⁺, with hydrated ionic radii of 3.67 Å and 4.94 Å respectively, so the corresponding flake size tends to be larger in the case of TMA⁺.³⁵ This trend can be seen from the dynamic light scattering (DLS) measurements shown in FIG. 2d, and additionally, DLS reveals that the mixed acid etch results in larger flake sizes compared to HF etching.

[0056] To study optical properties of the MXene colloids, UV-Vis spectra were collected from TBA-V₂CT_x (HF and mixed acid) and TMA d -V₂CT_x (mixed acid) colloids and are shown in FIG. 2e. The spectra for the HF etched sample shows a plateau in the low UV region (<250 nm), as seen in literature previously.⁴ Without being bound to any particular theory, this peak indicates the degradation of d -V₂CT_x. The lack of such a peak or plateau in the mixed acid etched samples is an indicator that the mixed acid etch results in flakes with higher quality with minimal content of oxidized/degraded flakes immediately after synthesis. Binder free, freestanding flexible MXene films with a golden-brown color (shown in FIG.s 3a,b) were obtained by vacuum filtering (<20 mL) d -V₂CT_x dispersed in water on a Celgard 3501 (0.22 μm pore size, 40 mm diameter) membrane. The SEM image in FIG. 3c shows the cross section of a TMA-V₂CT_x vacuum filtered film with a typical layered stack of individual MXene flakes. Corresponding XRD patterns of d -V₂CT_x films are shown in FIG. 3d. The $d_{(002)}$ -spacing of films prepared from TMA-V₂CT_x solutions (11.6 Å (2θ of 7.6°)) is smaller than TBA-V₂CT_x due to the smaller intercalant size. The TBA-V₂CT_x films from the HF etch and mixed acid etch show larger $d_{(002)}$ -spacings of 12.4 Å (2θ of 7.1°) and 14.8 Å (2θ of 5.95°), respectively. Without being bound to any particular theory, this variation could be the result of flake size difference, remaining stacks of few-layer flakes in the HF sample or an extra layer of water trapped in between the layers in the mixed acid sample.

[0057] As reported previously,^{17,26} V₂CT_x is unstable once delaminated. The rapid degradation of d -V₂CT_x in water dispersions was studied using UV-Vis. The complete oxidation of TBA-V₂CT_x (HF etched stored at 0.5 mg/mL) is shown in FIG. 4a,

with a color change from green-blue to yellow in the solution. The appearance of a second low-UV peak below 250 nm, and a broad decrease in the intensity in the 500-1000 nm wavelength region further suggests the presence of vanadium oxide species in the oxidized solution.²² FIG. 4b shows the transformation of the spectra for dilute (0.1 mg/mL) TBA- V_2CT_x synthesized from the mixed acid etch (the same style graph is shown for the HF etch in FIG. 11). The degradation begins immediately, with clear increases in the low UV region and reduction in the ~ 275 nm V_2CT_x peak, making the HF etched sample completely unusable after 3 hours and the mixed acid etch after 4 hours. It is worth noting that a 1-hour enhancement at such a dilute colloid may provide greater stability enhancements at higher concentrations.²⁹ This severe degradation is further shown in FIGs. 4c-e using films of TMA- V_2CT_x (mixed acid etch), vacuum filtered from fresh colloid, a one-month-old colloid (stored in argon filled vial in refrigerator), and a film prepared from a fresh colloid that was then left in the open air for one month. In all cases the films appear darker (compared to the brownish gold color for fresh films), but the oxidation in air was more detrimental as seen by the color and flexibility. This degradation can also be seen from the decrease in electrical conductivity of the films after oxidation. The film in FIG. 4c exhibited a conductivity of 648 S cm^{-1} , while the films in 4d and 4e had conductivities of 80 and 58 S cm^{-1} , respectively.

[0058] In order to overcome the rapid degradation and instability d - V_2CT_x , an ion-exchange process was used to replace the residual TBA^+ or TMA^+ with smaller alkali cations. It is worth noting that, in our experience, delamination with large organic TBA^+ or TMA^+ molecules is one of the culprits for defect formation in V_2CT_x flakes, the effect which may be more significant than harsh etching conditions. Therefore, development of etching and delamination procedures that eliminate the need for organic molecules and instead enable use of Li^+ (or other alkali ions) will be beneficial. FIG. 5a schematically illustrates the ion exchange and flocculation processes used to remove residual organic intercalants, substitute them with Li cations, and redisperse d - V_2CT_x solutions that now contain Li residuals.

[0059] The ion exchange process removes adsorbed ions from the MXene surface (TBA^+ , TMA^+) and replaces them with more desirable ions. This also results in tuning the MXene properties by changing their interlayer spacing and chemistry. An ion exchange process in multilayer $Ti_3C_2T_x$ MXene was previously demonstrated.³⁴ In conjunction with the ion exchange process, flocculation occurs due to the electrostatic

adsorption of cations on the negatively charged MXene surface. Previous reports of the zeta potential (surface charge) of HF etched d -V₂CT_x showed an average zeta potential of -32.4, which is close to the limit of colloidal stability (-30 mV).⁴ Using the mixed acid etch, average zeta potentials of -41.8 and -52.0 mV were achieved for TBA⁺ and TMA⁺ delaminated V₂CT_x respectively, which is shown in FIG. 12. With the addition of excess cations, flakes begin to crumple and restack in order to compensate the charge of the positive ion on the negatively charged flakes, and the flocculated flakes precipitate out of solution. Flocculation of Ti₃C₂T_x and V₂CT_x have been demonstrated using acids and bases, such as HCl and NaOH, as well as some alkali salts.^{36–38}

[0060] In order to perform ion-exchange and flocculation on TBA- or TMA-V₂CT_x colloids, a saturated LiCl water dispersion (19.8 M) is used to exchange TMA⁺/TBA⁺ for Li⁺. In a typical process, a 1:5 volume ratio of 19.8 M LiCl solution to MXene solution (concentration of ~0.5-1.0 mg/mL) was used. It is important to perform this process immediately after fresh d -V₂CT_x solutions are prepared to avoid degradation. After the sample has been flocculated, it should be left to naturally settle before any further handling. After the flocs have completely settled, if the sample is going to be kept for long-term storage (more than two weeks), the supernatant, which contains TMA⁺/TBA⁺, H₂O, and excess Li⁺, should be removed and replaced with 19.8 M LiCl solution. This can be accomplished using a pipette to remove the clear supernatant over the the flocculated MXene, followed by replacing the same volume with 19.8 M LiCl. This maximizes the LiCl: water ratio, minimizing hydrolysis.³⁹ Centrifugation may yield the same result, but there may be a difference in the structure and stacking of the centrifuged flocs, which may affect redispersability. This process allows d -V₂CT_x to be kept in a wet flocculated state (in a fridge) for storage until use. The flocculated V₂CT_x can be redispersed by washing with DI water and removal of excess Li ions (similar to washing after conventional intercalation/delamination). In short, the wet precipitates are transferred to centrifuge tubes and centrifuged at 3500 rpm (2550 rcf) for 10 minutes in order to ensure sedimentation of the flocculated MXene. The supernatant is decanted, and the washing process is continued by redispersing the precipitates in DI water, followed by hand shaking for 2 minutes between cycles. This is typically repeated 3-5 times, using ~750 mL of DI water for 1 g of V₂CT_x. Afterwards, the V₂CT_x flakes start to redisperse in water (supernatant becomes green/blue), and the supernatant can be collected. The obtained solutions are labeled as Li-V₂CT_x. The flocculated V₂CT_x solutions are shown in

FIG. 5b. For comparison, Li- V_2CT_x was prepared from TBA- V_2CT_x (HF and mixed acid etch) as well as TMA- V_2CT_x (mixed acid etch), and XRD analysis was performed on the vacuum filtered films. There is a clear and distinct shift in the position of (00 l) basal planes of V_2CT_x toward higher Bragg angles (smaller d -spacing) after ion-exchange, which indicates substitution of the majority of large TBA^+/TMA^+ ions with smaller Li^+ in the interlayer space. Moreover, there is a negligible difference in the $d_{(002)}$ -spacing (0.2 Å) of the Li- V_2CT_x films obtained from TMA or TBA- V_2CT_x solutions (FIG. 5c), which indicates that the ion exchange process can be done efficiently on both of these intercalants. The removal of residual TBA^+ and TMA^+ during the ion-exchange process was further investigated by using thermogravimetric analysis (TGA) as shown in figure 13a-e. Both TBA and TMA delaminated V_2CT_x samples showed a notable weight loss at ~400 °C (figure 13 b and c) which is attributed to the decomposition and removal of residual intercalants (TBA^+ or TMA^+).⁴⁰ However, similar to mI - V_2CT_x , the TGA analysis of Li ion-exchanged V_2CT_x samples did not show a weight loss peak in this temperature range, suggesting removal of residual TBA and TMA intercalants in these samples and high efficiency of the ion-exchange process.

[0061] In addition, compared to the previous report on ion-exchanged Li- V_2CT_x vacuum filtered films, our optimized process results in a more efficient removal of residual TBA^+ or TMA^+ .¹⁹ This can be seen from the obtained d -spacing values, where our Li- V_2CT_x films (prepared from TBA delaminated solutions) show a $d_{(002)}$ of ~10.0 Å whereas a value of 12.2 Å was reported in the previous work.¹⁹ FIG. 5d shows the conductivities of the V_2CT_x films obtained from vacuum filtration of different V_2CT_x solutions with different intercalants. The Li- V_2CT_x freestanding films exhibit electrical conductivity >1,000 S cm⁻¹ without annealing, roughly twice previous reports.²¹ The observed differences in the electrical conductivities of TBA or TMA- V_2CT_x and their corresponding Li- V_2CT_x films are likely due to differences in V_2CT_x flake size and quality. Larger V_2CT_x flake sizes in TMA intercalated films generally result in higher electrical conductivity compared to TBA intercalated films (a similar relationship has been reported for $Ti_3C_2T_x$).⁴¹ The decrease in the interlayer spacing of all Li- V_2CT_x films further facilitates inter-flake electron transport and, therefore, these films show the highest electrical conductivity. Li- V_2CT_x obtained from TMA- V_2CT_x solution shows a slightly higher conductivity as the result of its larger flake size. Additional factors that may influence the conductivity include the cations and water present in the interlayer of the

V_2CT_x , but these factors are complex and not currently well understood. The ion exchange and flocculation process can be performed with other ions, as Na^+ and Mg^{2+} have been demonstrated previously.¹⁹ In some cases, monovalent, high molarity salts (e.g., 30 m potassium acetate) can be used similarly.

[0062] The stability of the redispersed Li- V_2CT_x solutions was studied via UV-Vis as shown in FIG. 6a. TBA- V_2CT_x (HF etched) was kept in the flocculated state and redispersed after 33 and 147 days. Even after 147 days, the increase in the low UV region peak was minor and within the limits of what has been considered pristine d - V_2CT_x solutions in previous works.⁴ This 147-day old Li- V_2CT_x solution showed no visible signs of degradation, as seen in FIGs. 6b,c. The solution was subsequently vacuum filtered into a film that showed bright golden-brown color and good flexibility (FIG. 6d) in contrast to the TMA- V_2CT_x (FIG. 4d) which was prepared only after 30 days of storage. It is worth noting that the 102-day old sample had higher degradation than the 147-day old sample, which we assume is due to variation in the original samples. The same UV-Vis experiment was performed on Li^+ exchanged TMA and TBA- V_2CT_x (mixed acid etched) solutions, and the sample stability was checked at various times. Both mixed acid etched samples show no change in the UV-Vis spectra, indicating no degradation over time. This is in agreement with visual observations where the diluted redispersed Li- V_2CT_x solution showed no distinctive color change after 119 days and preserved its greenish blue color.

[0063] Solution Processing of Li- V_2CT_x

[0064] The improved quality and long-term stability of Li- V_2CT_x enables processing of these solutions in a variety of different ways, similar to $Ti_3C_2T_x$. As a proof of concept and for practicality and useability of the produced V_2CT_x solutions, we demonstrate a few common processing techniques to fabricate V_2CT_x materials with different forms and structures. FIGs. 7a,b show a 750 mL bottle of TMA- V_2CT_x solution and the flocculated Li- V_2CT_x , respectively. After redispersing, the Li- V_2CT_x solution was freeze-dried into an aerogel (FIG. 7c), which can be used for EMI shielding, adsorption, and electrochemical applications. The prepared Li- V_2CT_x solutions can be used to prepare hydrogels as well.

[0065] Hydrogels of varying transparency were prepared using mixtures of Li- V_2CT_x and PVA solutions in different ratios. The hydrogels (FIG. 7d) showed distinct optical properties and mechanical flexibility. They can be used in biomedical applications like physiological sensors and bioelectronic interfaces, as well as electrochemical

applications related to flexible, solid-state energy storage. Moreover, The V_2CT_x solutions can also be processed using spray coating. Transparent thin film V_2CT_x coatings were prepared by spray coating the Li- V_2CT_x solution with varying thicknesses onto glass slides (FIG. 7e). This is a relatively low-cost, simple method to generate transparent, conductive electrodes for electrochemical and optoelectronic applications. These examples demonstrate that the improved stability and properties of V_2CT_x MXene obtained through protocols and methods explained in this paper can open new pathways for practical use of V_2CT_x in wide-range of new applications.

[0066] Characterization Techniques and Procedure

[0067] A Zeiss Supra 50VP SEM was used for imaging of V_2AlC powder, multilayered and delaminated V_2CT_x , and vacuum filtered films. A Rigaku Smart Lab and powder diffractometer with a Cu $K\alpha$ target was used to collect XRD patterns from samples. MAX phase samples were scanned from $3-90^\circ 2\theta$, while MXene samples were scanned from $3-70^\circ 2\theta$. A current of 15 mA and a voltage of 40 kV were used with a step size of $0.02^\circ 2\theta$ and a duration time of 0.4 seconds. An Evolution 201 UV-Vis spectrophotometer (Thermo Scientific, MA, USA) with a 10 mm optical path length, quartz cuvette was used for analyses of optical properties. Spectra were collected from 200 to 1000 nm. Electrical conductivity of vacuum filtered V_2CT_x films and pressed multilayer V_2CT_x pellets were measured using a four-point probe conductivity measurement technique equipped with a 1 mm probe (Jandel Engineering Ltd., Bedfordshire, UK). The measured sheet resistance was converted to conductivity ($S\ cm^{-1}$) by factoring in the calculated thicknesses from either SEM or micrometer. Flake size and Zeta potential of solutions were estimated using a Malvern Panalytical Zetasizer Nano ZS in a folded capillary disposable cuvette. Five measurements were recorded in both cases, with the average being recorded.

[0068] Summary

[0069] In this disclosure, we show how to overcome stability limitations when working with V_2CT_x , which is known as one of the least stable MXenes. The viability of using a mixed acid etchant for MXene synthesis was shown. We developed an efficient and robust ion-exchange protocol that increases its chemical stability, both in wet and dry form. Most important, the stability of delaminated V_2CT_x MXene in aqueous solutions increased from a few hours to several months, opening new horizons for MXene applications. This ion exchange process enables tuning of the interlayer space, resulting in

an increased electrical conductivity of V_2CT_x free-standing films above all the values reported in the literature. We have demonstrated and discussed improved etching, delamination, and storage approaches and protocols for V_2CT_x . Therefore, this work can be looked at as a comprehensive guideline for synthesis of chemically stable MXene solutions (e.g., including V_2CT_x) and films with improved properties.

[0070] General Applicability of Mixed Acid Etch and Ion Exchange Method

[0071] The mixed acid etch described in the main text was performed on three different MAX phase precursors. The “Conventional MAX” was synthesized via the protocols described in the main text. As for the “High Al” MAX phase, the same synthesis conditions were used, but a higher aluminum content was introduced, similar to for the method described for $Ti_3C_2T_x$.¹ The “Carbon Ukraine MAX” sample was obtained from Carbon Ukraine, Ltd. The XRD patterns of the corresponding MAX phases are shown in FIG. 9a, demonstrating variations in the impurities present between the samples. These XRD patterns were collected after acid washing the samples according to the protocol described in the main text.

[0072] The different MAX phase precursors were all etched using the mixed acid etch, delaminated with TBAOH, and flocculated/exchanged with Li^+ to study the stability of the samples. In FIG. 9b, the UV-Vis spectra of the redispersed $Li-V_2CT_x$ samples from different MAX phase precursors are shown. The Conventional MAX phase and High Al sample show no signs of degradation, while the Carbon Ukraine sample shows minimal oxidation. The SEM images in FIGs. 9c-e show smoother particulates from the MAX phases synthesized in the lab compared to the milled commercial powder. The small particles in the Carbon Ukraine sample could be easier to oxidize. However, the UV-Vis spectra of the 104-day old Carbon Ukraine sample still only showed minimal oxidation. Thus, $Li-V_2CT_x$ MXenes prepared from all three precursors display enhanced stability. This demonstrates versatility and universal applicability of the developed process.

[0073] In order to further characterize the water and residual intercalant contents of the as-synthesized V_2CT_x as well as ion exchanged V_2CT_x , thermogravimetric analysis (TGA) was performed in an argon atmosphere on samples at different stages of synthesis and post-processing. In figure 10a, the TGA plot for *ml*- V_2CT_x is shown. FIGs. 10B and 10C show the TGA plots for TBA- and TMA- V_2CT_x , respectively. Also, FIGs. 10D and 10E show TGA plots for $Li-V_2CT_x$ made from TBAOH and TMAOH, respectively.

[0074] TGA data reveal more information about water content and residual TBA⁺ and TMA⁺ cations in different V₂CT_x samples before and after delamination as well as the efficacy of ion-exchange process. *ml*-V₂CT_x (FIG. 12A) shows a three-stage weight loss (within the temperature range up to ~700 °C) starting with an initial desorption of physisorbed water from ~50-225 °C. At higher temperatures (~380-600 °C) dissociation of surface functional groups occurs and also chemisorbed or structural water is removed from multilayer powders. V₂CT_x MXene starts to degrade at around ~ 600 °C in inert atmosphere.² The degradation becomes more significant as the temperature is increased. The weight loss starting at ~700 °C is due to degradation of MXene structure, as also previously shown for Ti₃C₂T_x.³

[0075] In contrast to *ml*-V₂CT_x, up to ~700 °C, TBA delaminated V₂CT_x shows a four-stage weight loss in its TGA pattern (FIG. 12b). A larger weight loss at ~60-200 °C followed by a notable weight loss at around ~250-320 °C can be attributed to removal of water from the structure. Without being bound to any theory, one may assume that TBA co-intercalates more water molecules during delamination, as also evident from its larger *d*₍₀₀₀₎-spacing. The third stage of weight loss can be seen at ~325-430 °C, which is due to the decomposition and removal of the residual TBA ions from MXene.³ A similar trend is observed for TMA delaminated V₂CT_x (FIG. 12c). However, in case of ion exchanged Li-V₂CT_x samples prepared from the TBA or TMA (FIG. 12d,e), the weight loss at low temperatures is less (similar to that of *ml*-V₂CT_x) and also the weight loss peak at ~325-430 °C is absent, which indicates that residual TBA or TMA cations are completely removed from the structure during the ion-exchange process. Similar to *ml*-V₂CT_x, but at slightly higher onset temperatures, both samples show the second stage weight loss at ~ 445 °C related to dissociation of surface functional groups and removal of chemisorbed water. These results further support the decreased *d*₍₀₀₀₎-spacing and increased electrical conductivity of Li-V₂CT_x films after ion-exchange process and shed light on the effectiveness of this process to remove residual TBA or TMA cations and exchange them with Li⁺ or other alkali cations.

Table 1. Etching Configurations Tests

Etching Conditions (Per 1 g V ₂ AlC) 12 mL (48%) HF: 8 mL (12M) HCl at 50 °C	Result
48 hours	Low yield, likely under-etched
60 hours	Yield below 50%, no initial oxidation
72 hours	Yield above 50%, no initial oxidation

Table 2. Nomenclature Guide

Abbreviation	Explanation
V_2CT_x	Vanadium carbide MXene
$ml-V_2CT_x$	Multilayered vanadium carbide MXene
$d-V_2CT_x$	Delaminated vanadium carbide MXene (general)
$TBA-V_2CT_x$	Vanadium carbide MXene delaminated with TBAOH
$TMA-V_2CT_x$	Vanadium carbide MXene delaminated with TMAOH
$Li-V_2CT_x$	Lithium ion exchanged with TBA- or TMA- V_2CT_x

Table 3. Chemical Suppliers List

Material/Chemical	Supplier
Vanadium (99.5%, -325 mesh)	Alfa Aesar
Aluminum (99.5%, -325 mesh)	Alfa Aesar
Graphite (99%, -325 mesh)	Alfa Aesar
9M HCl	Fischer Scientific, U.S.
48 wt.% HF	Acros Organics, Fair Lawn, NJ
25 wt.% TMAOH	Sigma Aldrich
40 wt.% TBAOH	Alfa Aesar

[0076] Aspects

[0077] The following Aspects are illustrative only and do not limit the scope of the present disclosure or the appended claims. Any one or more parts of any Aspect can be combined with any one or more parts of any other Aspect.

[0078] Aspect 1. A method, comprising: providing a solution of delaminated MXene material and a first ionic intercalant; and contacting the solution with a second ionic intercalant so as to exchange the first ionic intercalant and the second ionic intercalant so as to give rise to flocculated portions of the MXene material and a supernatant that comprises the first ionic intercalant and the second ionic intercalant.

[0079] Aspect 2. The method of Aspect 1, further comprising replacing at least some of the supernatant with a saturated solution of the second ionic intercalant.

[0080] Aspect 3. The method of Aspect 2, wherein the saturated solution of the second ionic intercalant is a supersaturated solution.

[0081] Aspect 4. The method of any one of Aspects 1-3, wherein the first ionic intercalant comprises an organic base, the organic base optionally comprising TMAOH, TBAOH, TEAOH, TPAOH or any combination thereof.

[0082] Aspect 5. The method of any one of Aspects 1-4, wherein the method of any one of Aspects 1-4, wherein the second ionic intercalant comprises an alkali cation-containing inorganic salt, the alkali cation-containing inorganic salt optionally comprising LiCl, NaCl, KCl, MgCl₂, CaCl₂, Li₂SO₄, K₂SO₄, MgSO₄, Na₂SO₄, LiOH, NaOH, KOH, or any combination thereof.

[0083] Aspect 6. The method of any one of Aspects 1-5, wherein the flocculated portions of MXene material are one or both of single layer MXene or few-layer MXene. (A few-layer MXene has from 2 to 5 atomic layers.)

[0084] Aspect 7. The method of any one of Aspects 1-7, wherein the MXene has the formula $M_{n+1}C_nT_x$, wherein M is V.

[0085] Aspect 8. The method of Aspect 8, wherein the MXene has the formula of V_2CT_x .

[0086] Aspect 9. The method of any one of Aspects 1-6, further comprising redispersing the flocculated MXene portions in solution.

[0087] Aspect 10. The method of Aspect 9, wherein the flocculated portions of MXene material remain essentially undegraded after storage under ambient conditions for 150 days.

[0088] Aspect 11. A MXene dispersion, comprising a plurality of portions of single-layer and/or few-layer MXene dispersed in a solvent, the solvent comprising a first ionic intercalant and a second ionic intercalant.

[0089] Aspect 12. The MXene dispersion of Aspect 11, wherein the first ionic intercalant comprises TMAOH and/or TBAOH.

[0090] Aspect 13. The MXene dispersion of any one of Aspects 11-12, wherein the second ionic intercalant comprises LiCl.

[0091] Aspect 14. The MXene dispersion of any one of Aspects 11-13, wherein the MXene has the formula $M_{n+1}C_nT_x$, wherein M is V.

[0092] Aspect 15. The MXene dispersion of Aspect 14, wherein the MXene has the formula of V_2CT_x .

[0093] Aspect 16. A stable MXene dispersion, comprising a plurality of portions of single-layer and/or few-layer MXene dispersed in a solvent, the solvent comprising an ionic intercalant, the ionic intercalant optionally being present in supersaturated form.

[0094] Aspect 17. The stable MXene dispersion of Aspect 16, wherein the ionic intercalant comprises LiCl.

[0095] Aspect 18. The stable MXene dispersion of any one of Aspects 16-17, wherein the MXene has the formula $M_{n+1}C_nT_x$, wherein M is V.

[0096] Aspect 19. The stable MXene dispersion of Aspect 18, wherein the MXene has the formula of V_2CT_x .

[0097] Aspect 20. The stable MXene dispersion of any one of Aspects 16-19, wherein the MXene remains essentially undegraded after storage under ambient conditions for 100 days.

[0098] References

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[00140]

What is Claimed:

1. A method, comprising:

providing a solution of delaminated MXene material and a first ionic intercalant; and

contacting the solution with a second ionic intercalant so as to exchange the first ionic intercalant and the second ionic intercalant so as to give rise to flocculated portions of the MXene material and a supernatant that comprises the first ionic intercalant and the second ionic intercalant.
2. The method of claim 1, further comprising replacing at least some of the supernatant with a saturated solution of the second ionic intercalant.
3. The method of claim 2, wherein the saturated solution of the second ionic intercalant is a supersaturated solution.
4. The method of any one of claims 1-3, wherein the first ionic intercalant comprises an organic base, the organic base optionally comprising TMAOH, TBAOH, TEAOH, TPAOH or any combination thereof.
5. The method of any one of claims 1-3, wherein the method of any one of claims 1-4, wherein the second ionic intercalant comprises an alkali cation-containing inorganic salts, the alkali cation-containing inorganic salt optionally comprising LiCl, NaCl, KCl, MgCl₂, CaCl₂, Li₂SO₄, K₂SO₄, MgSO₄, Na₂SO₄, LiOH, NaOH, KOH, or any combination thereof.
6. The method of any one of claims 1-3, wherein the flocculated portions of MXene material are one or both of single layer MXene or few-layer MXene.
7. The method of any one of claims 1-3, wherein the MXene has the formula $M_{n+1}C_nT_x$, wherein M is V.
8. The method of claim 8, wherein the MXene has the formula of V_2CT_x .
9. The method of any one of claim 1-3, further comprising redispersing the flocculated MXene portions in solution.

10. The method of claim 9, wherein the flocculated portions of MXene material remain essentially undegraded after storage under ambient conditions for 150 days.
11. A MXene dispersion, comprising:

a plurality of portions of single-layer and/or few-layer MXene dispersed in a solvent, the solvent comprising a first ionic intercalant and a second ionic intercalant.
12. The MXene dispersion of claim 11, wherein the first ionic intercalant comprises TMAOH and/or TBAOH.
13. The MXene dispersion of any one of claims 11-12, wherein the second ionic intercalant comprises LiCl.
14. The MXene dispersion of any one of claims 11-12, wherein the MXene has the formula $M_{n+1}C_nT_x$, wherein M is V.
15. The MXene dispersion of claim 14, wherein the MXene has the formula of V_2CT_x .
16. A stable MXene dispersion, comprising

a plurality of portions of single-layer and/or few-layer MXene dispersed in a solvent, the solvent comprising an ionic intercalant, the ionic intercalant optionally being present in supersaturated form.
17. The stable MXene dispersion of claim 16, wherein the ionic intercalant comprises LiCl.
18. The stable MXene dispersion of any one of claims 16-17, wherein the MXene has the formula $M_{n+1}C_nT_x$, wherein M is V.
19. The stable MXene dispersion of claim 18, wherein the MXene has the formula of V_2CT_x .
20. The stable MXene dispersion of any one of claims 16-17, wherein the MXene remains essentially undegraded after storage under ambient conditions for 150 days.

FIG. 1A

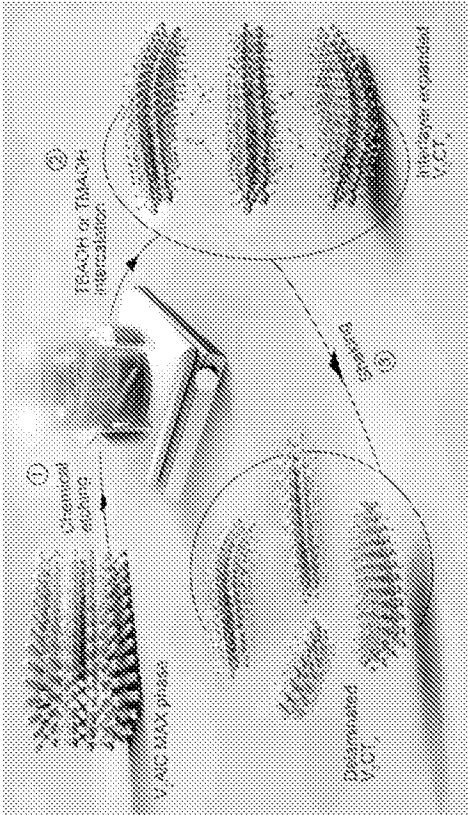


FIG. 1B

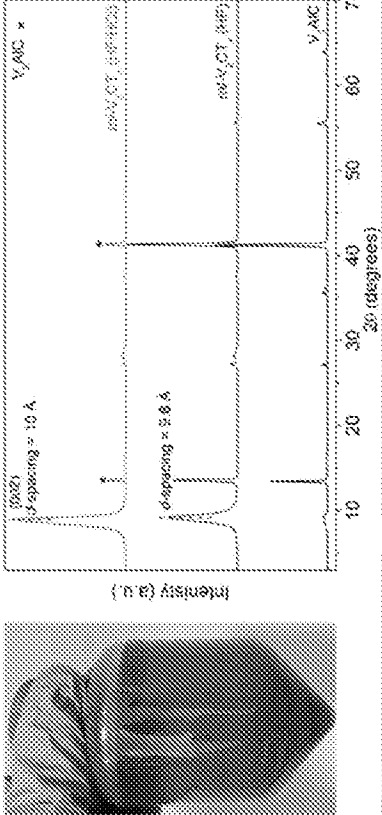


FIG. 1C

FIG. 1D

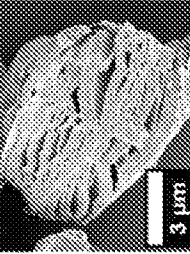


FIG. 1E

FIG. 1F

FIG. 2A

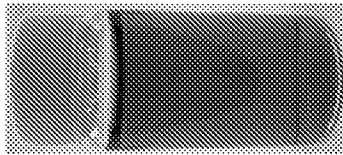


FIG. 2B

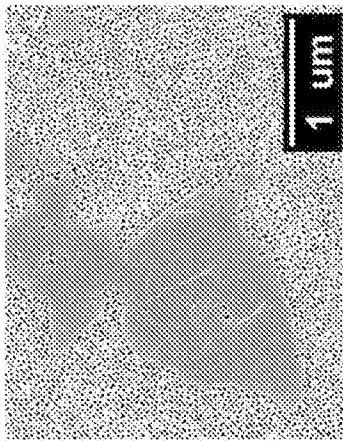


FIG. 2C

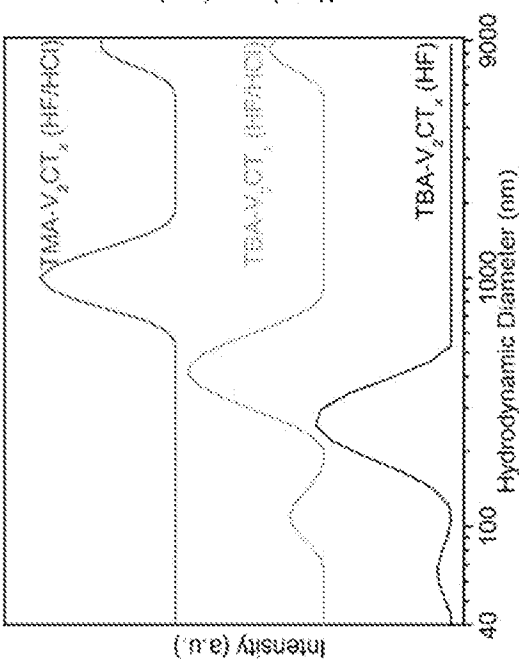
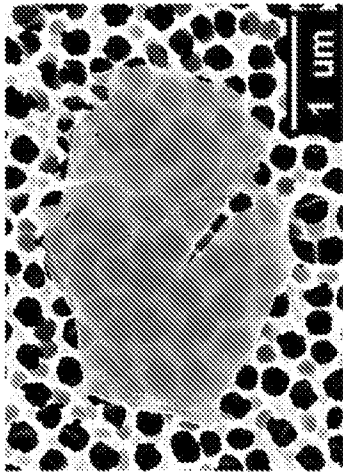


FIG. 2D

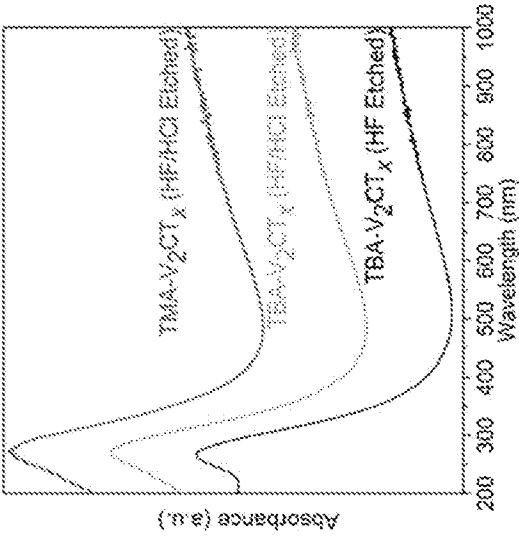


FIG. 2E

FIG. 3C

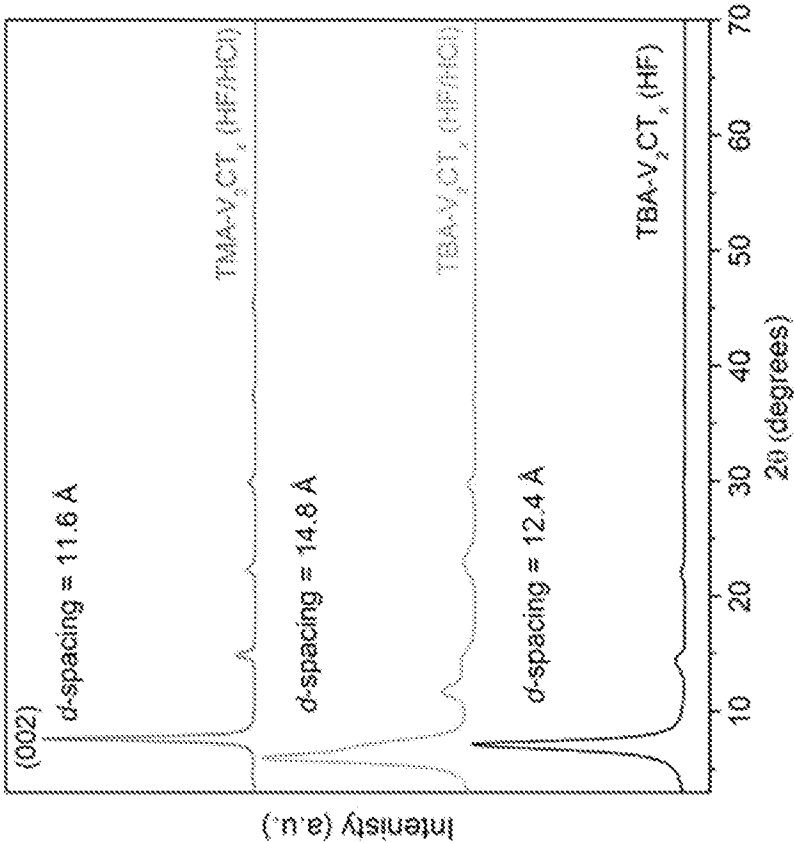


FIG. 3B

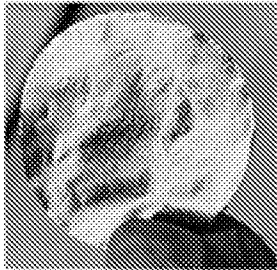


FIG. 3A

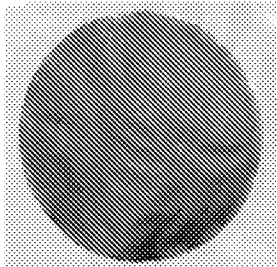


FIG. 3D

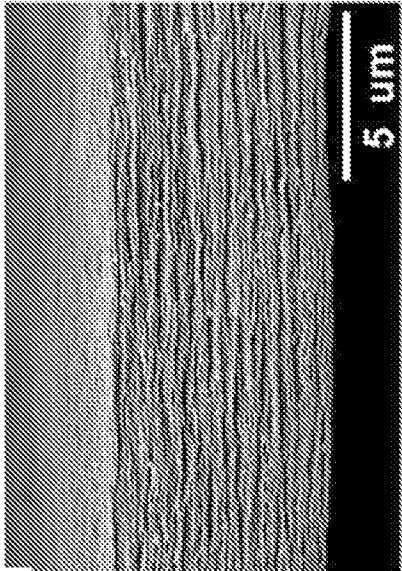


FIG. 4A

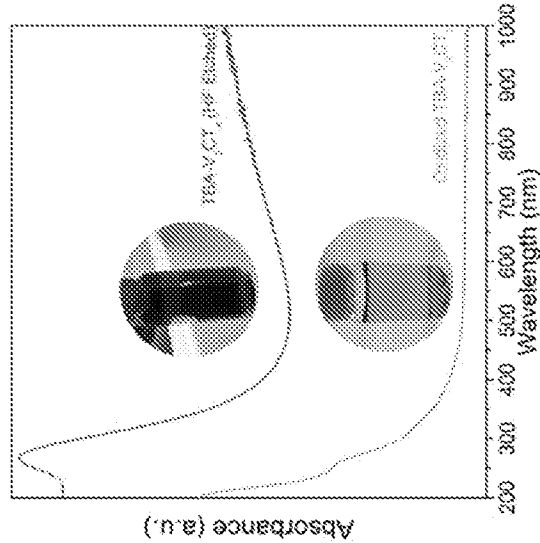


FIG. 4B

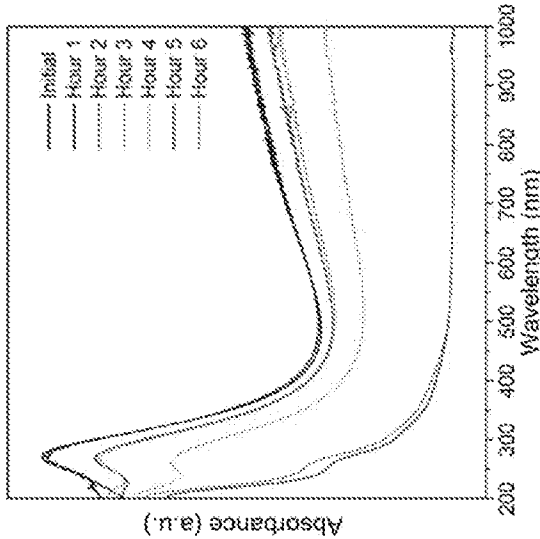


FIG. 4C

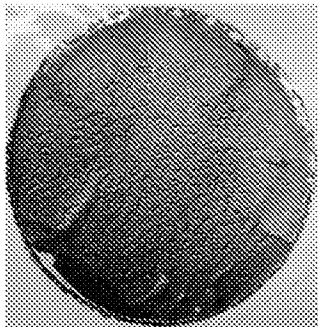


FIG. 4D

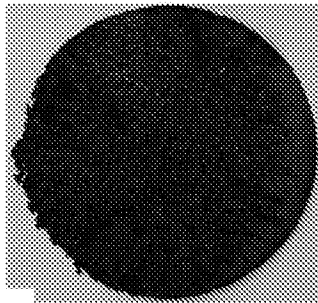


FIG. 4E

FIG. 5A

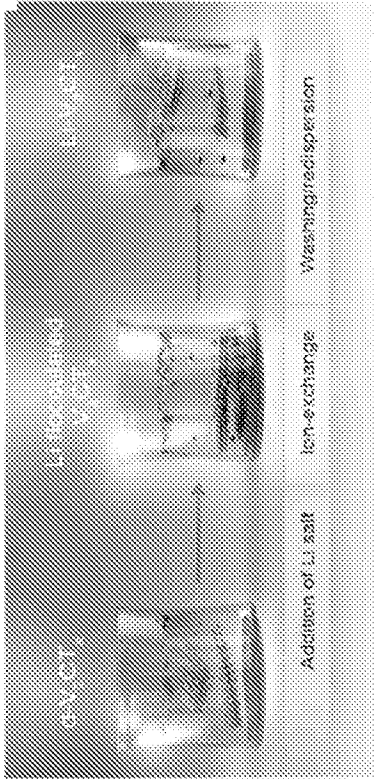


FIG. 5B

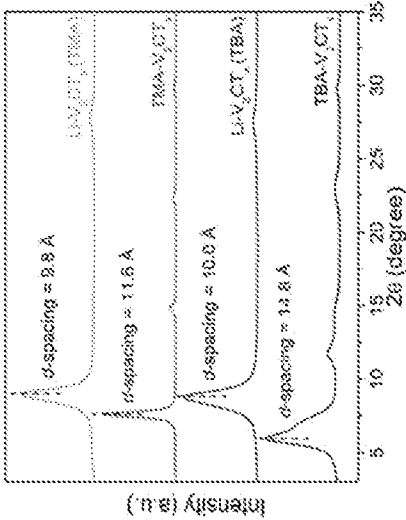


FIG. 5C

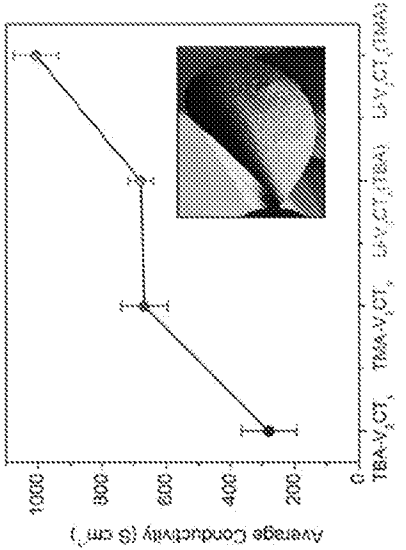


FIG. 5D

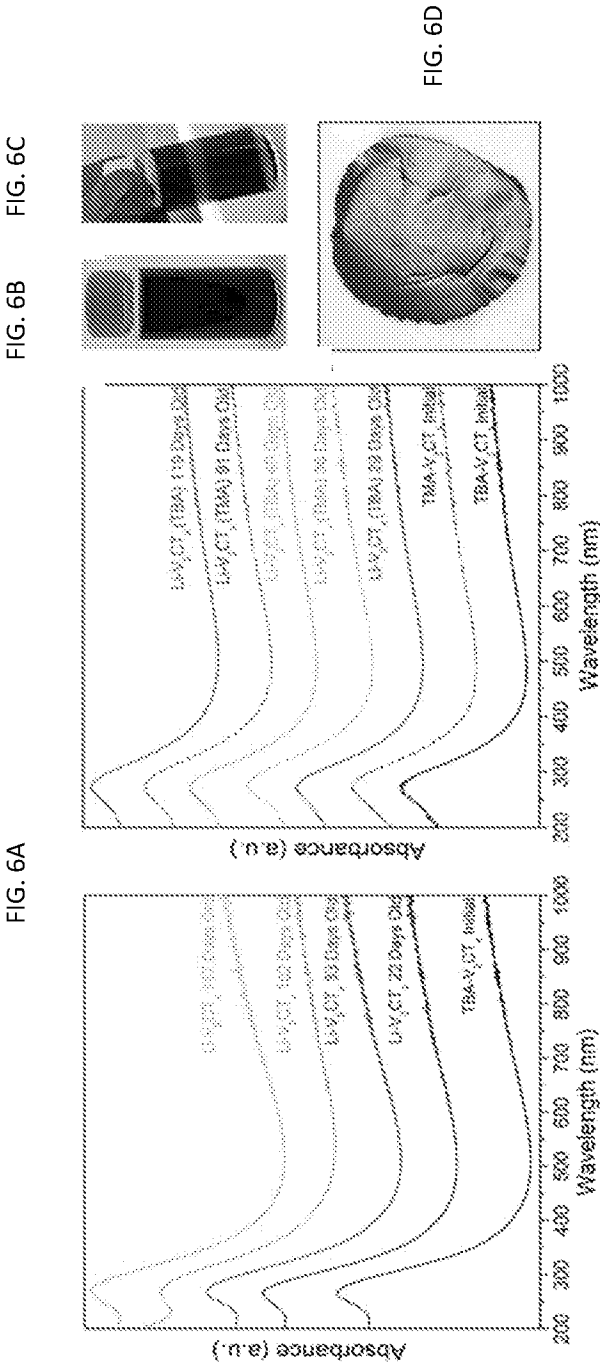


FIG. 7A

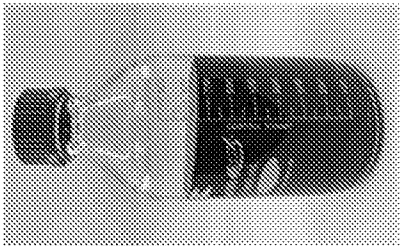


FIG. 7B

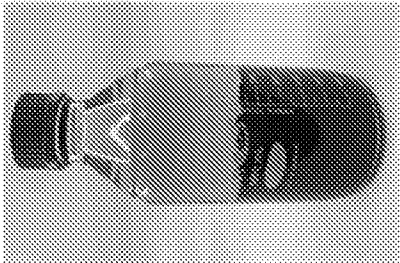


FIG. 7C

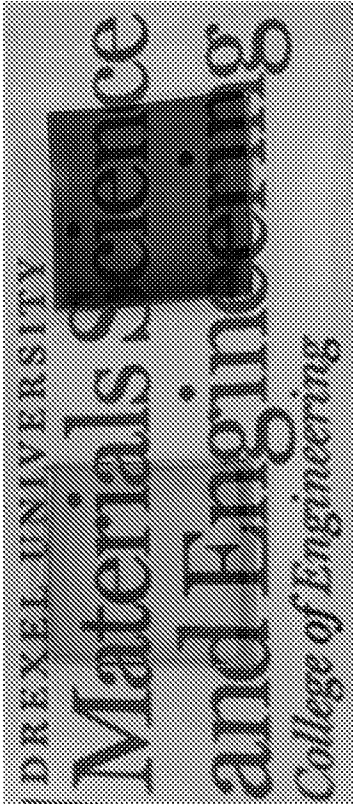
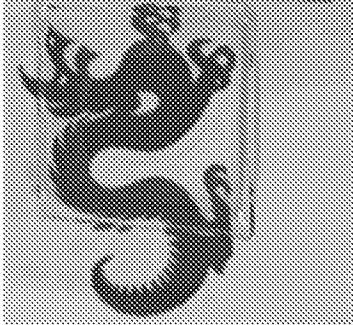
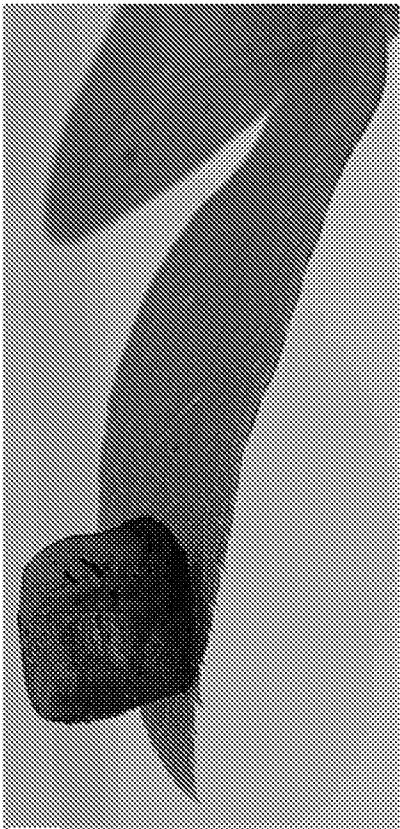


FIG. 7D

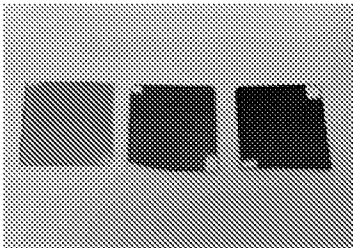


FIG. 7E

Synthesis of V_2CT_x MXene

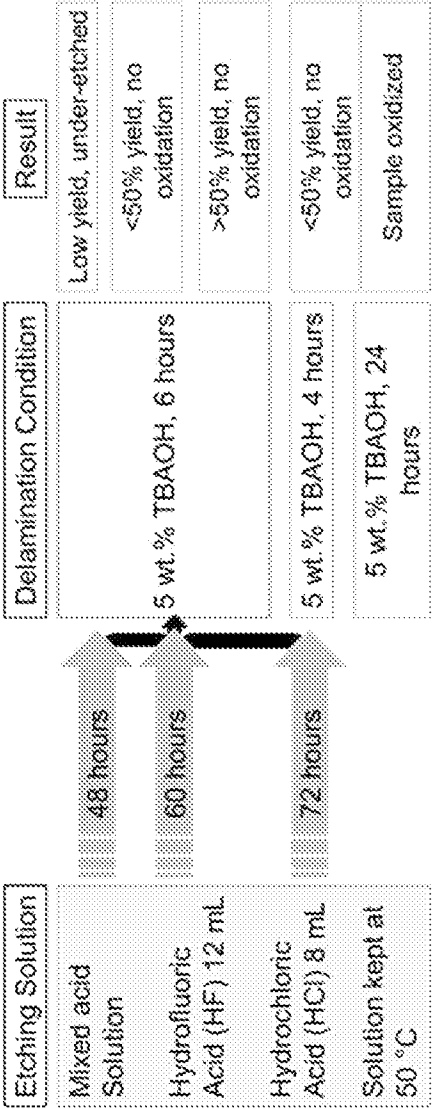


FIG. 8

FIG. 9C

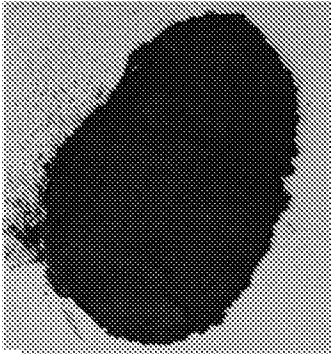


FIG. 9B

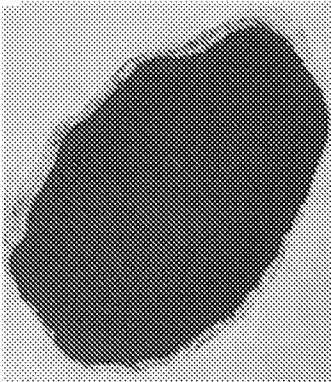


FIG. 9A



FIG. 9E

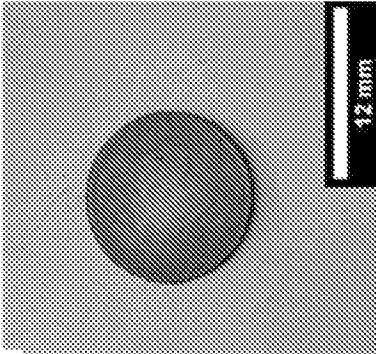


FIG. 9D

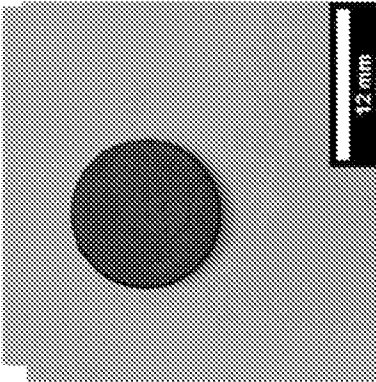


FIG. 10A

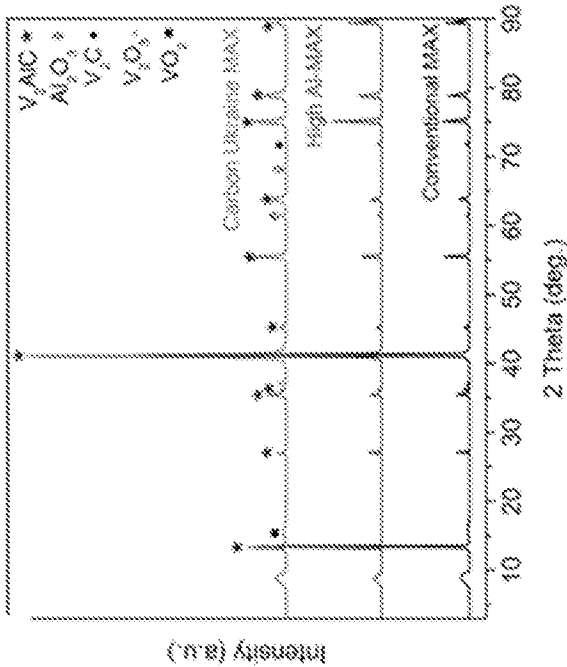


FIG. 10B

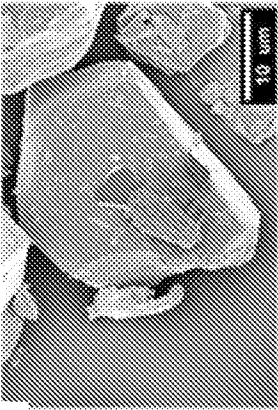
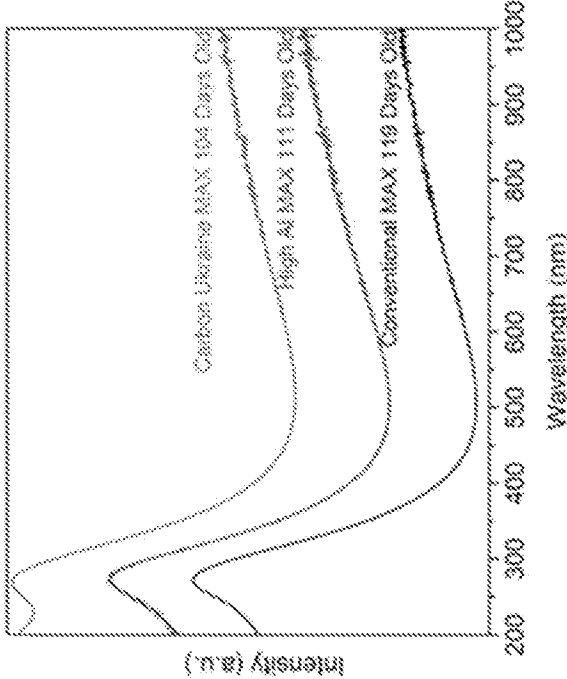


FIG. 10C

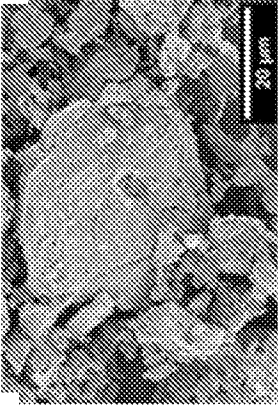


FIG. 10D

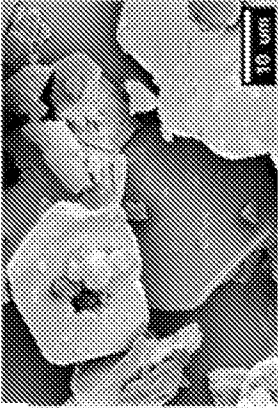


FIG. 10E

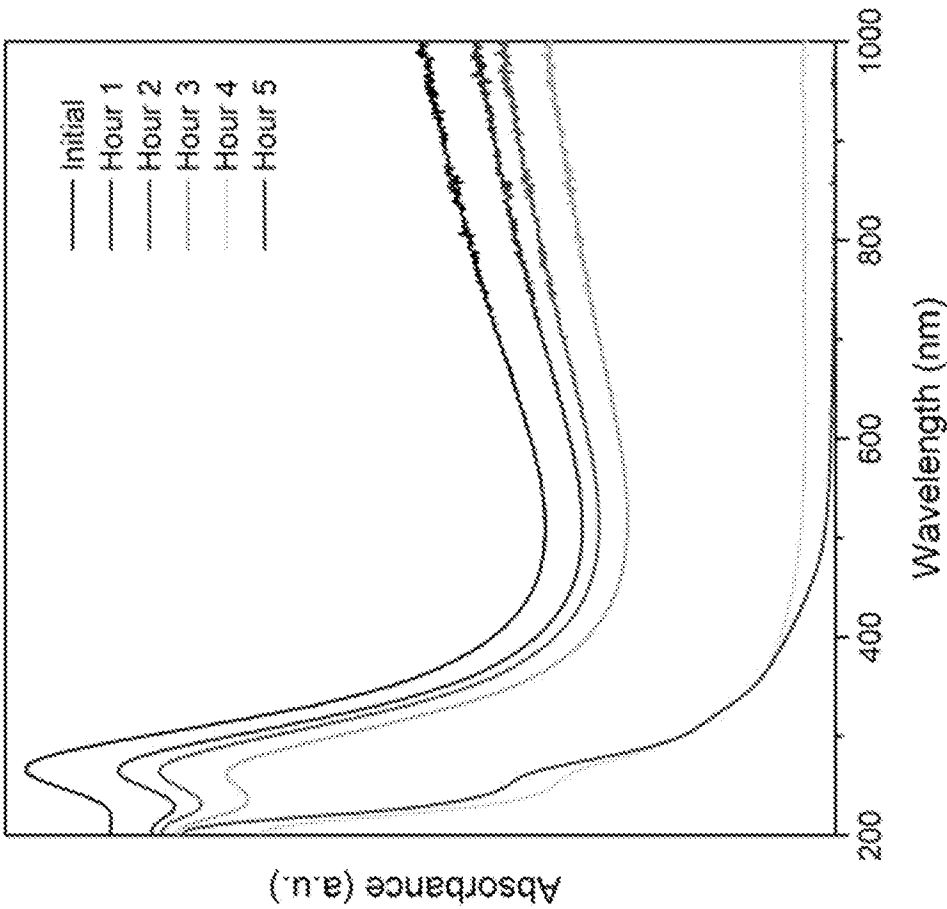


FIG. 11

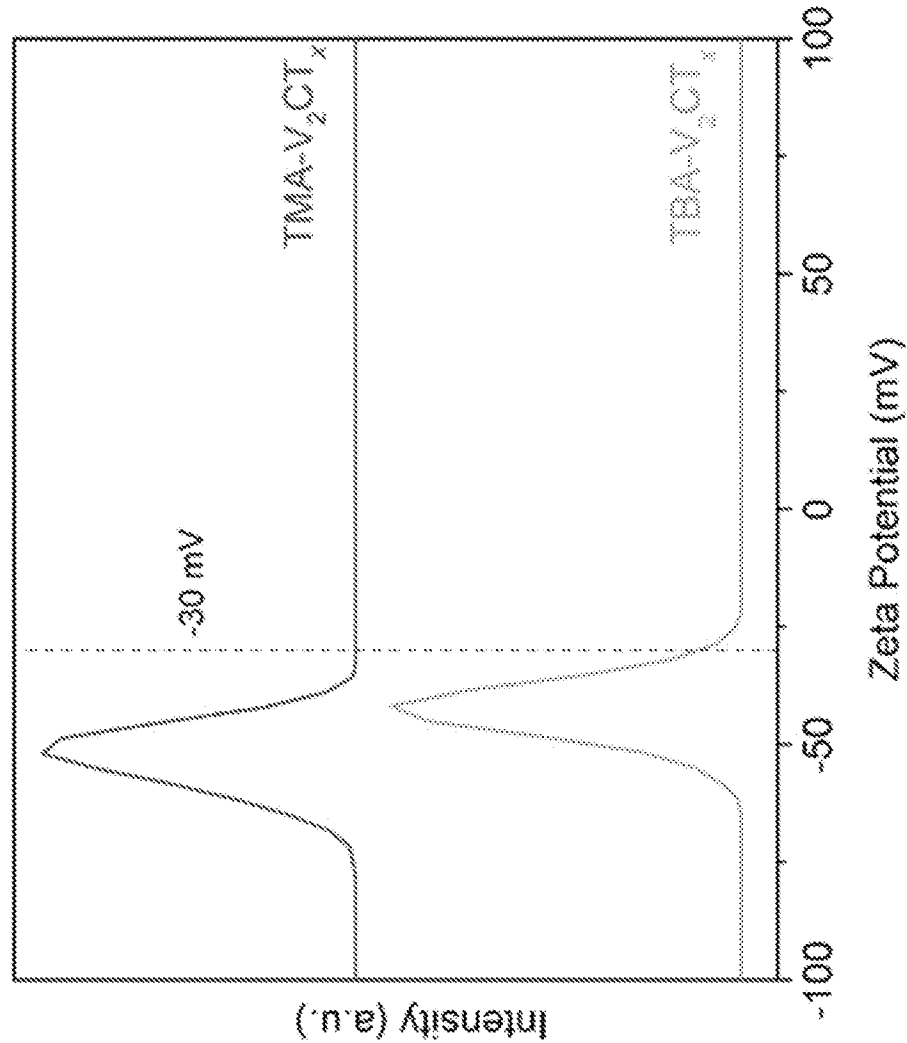


FIG. 12

FIG. 13A

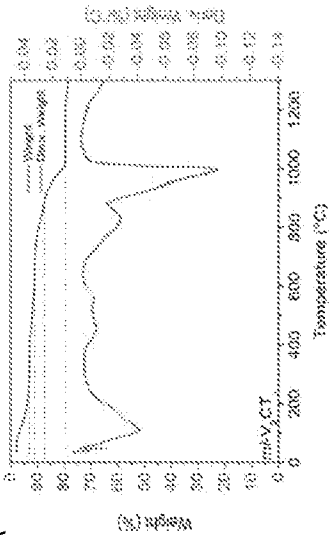


FIG. 13B

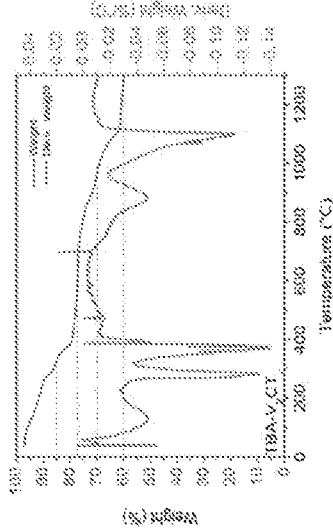


FIG. 13C

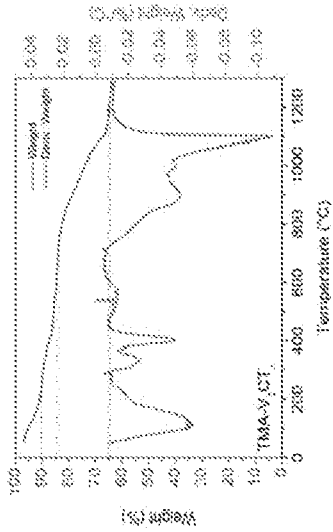


FIG. 13D

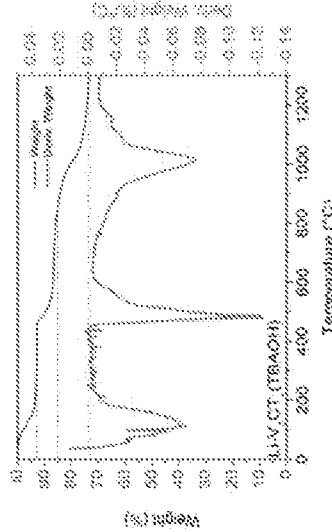


FIG. 13E

