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Morphological, Structural and Radon Concentration Properties of Two-Dimensional Titanium Carbide MXene, Ti_3C_2

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ABSTRACT. In this study, radon concentration of 2D Titanium Carbide MXene Ti_3C_2 was studied by using an established radon monitor. A layered MAX phase of Ti_3AlC_2 were synthesized through pressureless sintering (PLS) by the initial powder of TiH_2/Al/C without preliminary dehydrogenation under argon atmosphere at 1350 °C. An elegant exfoliation approach was used to eliminate Al from its precursor to form a layered-structure of Ti_3C_2. Morphological and structural properties of this 2D material also studied. SEM images show two types of morphology which is a layer of Ti_3C_2 and the agglomerates Al_2O_3 with graphite. XRD pattern reveals three phases in this material which is a rhombohedral Al_2O_3, rhombohedral graphite and rhombohedral Ti_3C_2 phases. Radon concentration for this material for five consecutive days explains the safety used of this material is under the maximum value from US Environmental Protection Agency (EPA).

Keywords: *MAX phase, MXene, Two-dimensional materials, Ti_3C_2, Radon concentration;*

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1. INTRODUCTION

Two-dimensional (2D) materials are materials with unique properties which can be defined as a material with single atomic plane, such as graphene, with have only 0.34 nm thick of one atomic layer of carbon [1]. The range of materials to be called as 2D materials are from 1 layer to 10 layers, where more than 10 layers is considered as 3D materials [2]. By far the most studied 2D material is graphene, which is comprised of atomically thin layers of sp^2-bonded carbon atoms connected by aromatic in-plane bonds. Since the outstanding electronic properties of graphene have been discovered, other alternative 2D materials have attracted much renewed attention.

A fascinating idea was proposed to prepared freestanding graphene-like carbides (and nitrides) from ternary layered MAX phases (known also as nanolaminates) [3], which includes various 2D-like layers of transition metal carbides (or nitrides) as building blocks [4]. These 2D materials are now known as 'MXenes'; this term denotes their genesis from MAX phases (with the loss of the A component) and their similarity to graphene [5].

MXenes is new 2D nanosheet materials and gaining so much attention from materials engineer because its properties and unique behaviour and has similar lamellar structure with graphene.

Generally, a 2D material is produced by removing A from MAX phases by chemical etching. MAX phases are ternary carbide or nitrides with the chemical formula of $M_{n+1}AX_n$, where M is an early transition metal, A is an A-group element and X is either carbon or nitrogen. The value of n can be 1, 2 or 3 [3]. There is no research until recently was discussed about the radon-222 (^{222}Rn) concentration of MXene, and it is important to study since long term exposure to radon is associated with lung cancer risk and present a significant environmental health hazard. ^{222}Rn is the heaviest gas element, nine times heavier than air and a noble radioactive gas. This element does not chemically react with other materials in air and was occurs naturally from U-238 decay process. Inhalation of ^{222}Rn and ^{222}Rn daughter products for human will increase the possibility of lung cancer due to the presence of alpha (α) particles which have linear energy transfer (LET) and that affects the alveolus [6]. Measurements of radon are normally expressed as the concentration of radon in units of picocuries per liter of air (pCi/L) and the United States EPA recommendations are about 4 pCi/L or lower, which is the average or slightly above average for safety requirement in any applications [7].

The aims of this research are to study the significant radon concentration of 2D MXene Ti_3C_2 for future application in engineering and technology. In addition, morphological and structural analysis was also performed to investigate the microstructural and elemental properties of 2D MXene Titanium Carbide Ti_3C_2.

2. MATERIALS AND METHODS

Starting materials of titanium hydrate (TiH_2, 99%, 325 mesh, Alfa Aesar, United State), aluminium (Al, 99%, 200 mesh, Acros Organics, Austria) and graphite (C, 99%, 550 mesh, Acros Organics, Germany) were used in this research. The powder was weighed according to the stoichiometry ratio of 3:1.1:2, then mixed by using a planetary ball mill for 60 hours. The powder was then cold-pressed to form a pellet with diameter of 1 cm and thickness of 0.3 cm before sintered in tube furnace by using a pressureless sintering (PLS) technique. The heating rate was controlled at 10 °C/min and the sintering temperature was selected at 1350 °C for 2 hours in argon atmosphere. The pellet then was pulverized, immersed and stirred in hydrofluoric solution (HF, AR grade, 49%, Qrec, New Zealand) for 20 hours in fume hood. The resulted mixture was then placed in centrifuge tube and centrifugation process was run at 5000 rpm for 7 min. The mixture was then washed with deionized water for several times, until the pH of solution approaching 7. The resulting Ti_3C_2 was then dried in vacuum at 50 °C for 24 hours.

X-ray diffraction (XRD) analysis was performed using Bruker D2 Phaser at 2θ values of 20 - 60° with Cu Kα radiation to determine the corresponding peaks of the MXene. X'pert HighScore Plus software was used to match the corresponding peaks with the standard data from International Committee of Diffraction Data (ICDD) X-ray data file. Microstructure analysis was performed using Nova NanoSEM 450 to characterize surface morphology of MXene Ti_3C_2.

A pellet of MXene Ti_3C_2 together with the professional radon monitor model 05-240, thermometer and hygrometer were placed in closed, fabricated container to calculate the radon concentration by time. The small openings of the container were sealed by using modelling material. The build-up of Radon (Rn) inside the container was measured using the Rn monitor. Rn concentration (pCi/L) was recorded every hour for five consecutive days.

3. RESULTS AND DISCUSSION

The morphology of 2D MXene Ti_3C_2 shows the layered structure with the thickness of 1 μm due to the removal of Al during elegant exfoliation approach in 49% HF solution (Fig. 1). SEM images shows two types of morphology which are a layer of Ti_3C_2 (point A) and the agglomerates Al_2O_3 with graphite (point B). The images of layered Ti_3C_2 with the thickness of 0.1-1.0 μm confirms the successful exfoliation of Ti_3C_2 similar with reported exfoliation graphene [8]. Sonication of HF treated resulted the separation of 2D Ti_3C_2 MXene.

The agglomerates of Al_2O_3 and graphite may formed during sintering and because of the stability of this phase, it is impossible to etched during elegant exfoliation process [9].

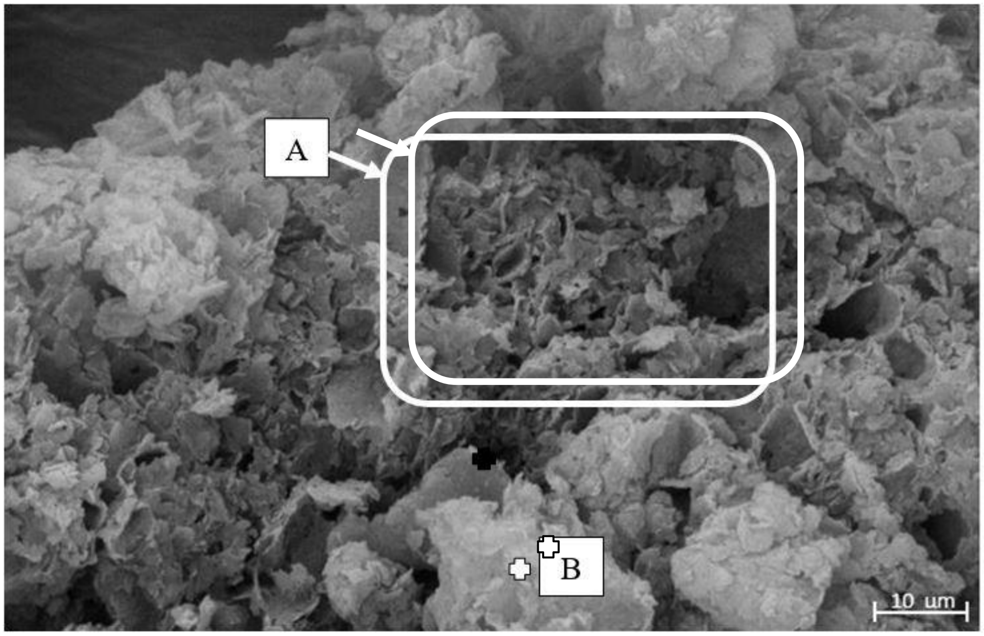

Fig. 1 SEM image of 2D MXene Ti_3C_2 after etching process in 49% HF solution

The structural analysis of 2D MXene Ti_3C_2 shows there are three phases matches which is rhombohedral Al_2O_3, rhombohedral graphite and rhombohedral Ti_3C_2 phases (Fig. 2). This XRD result is also supported by other researchers [9-15] in this field based on the reactions of the HF solutions with Ti_3AlC_2 which include:

$$Ti_3AlC_2 + 3HF \rightarrow AlF_3 + 3/2H_2 + Ti_3C_2 \quad (1)$$

$$Ti_3C_2 + 2H_2O \rightarrow Ti_3C_2(OH)_2 + H_2 \quad (2)$$

$$Ti_3C_2 + 2HF \rightarrow Ti_3C_2F_2 + H_2 \quad (3)$$

The formation of Ti_3C_2 phase in XRD pattern instead of $Ti_3C_2(OH)_2$ in reaction (2) was attributed to the ultrasonication in methanol leads to exfoliation of MXene Ti_3C_2.

The radon concentration properties of 2D MXene Ti_3C_2 shows the rate of radon was slightly increase started from day 2 to day 5 (Fig. 3). Shaded region shows the concentration level is in between of maximum and minimum values for each days. The highest radon concentration was recorded at day 5 (1.2 pCi/L) which is lower that the suggested value by United State Environmental Protection Agency (EPA). Hence, it was experimentally proven that Ti_3C_2 could be an alternative material towards the development of new electronic technologies due to small radiation emission and considered safe for consumer usage.

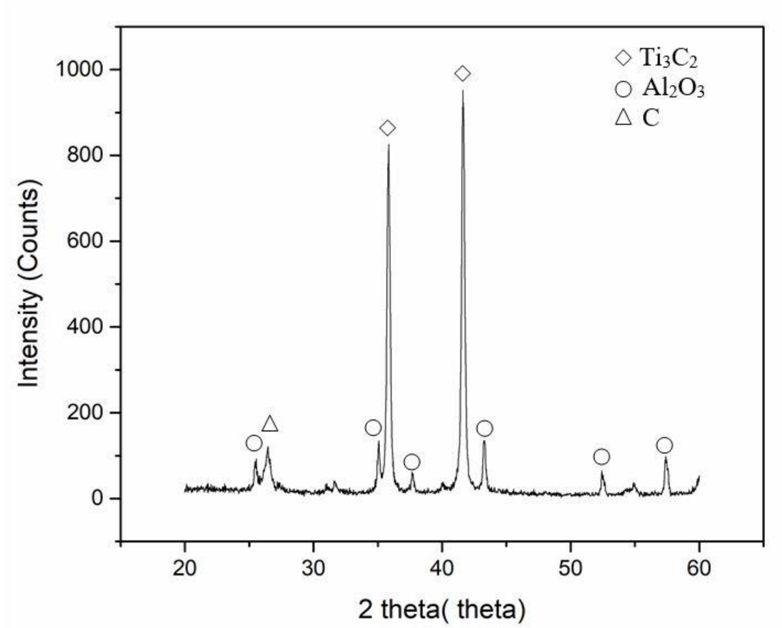

Fig. 2 XRD pattern of 2D MXene Ti_3C_2 after etched in 49% HF solution

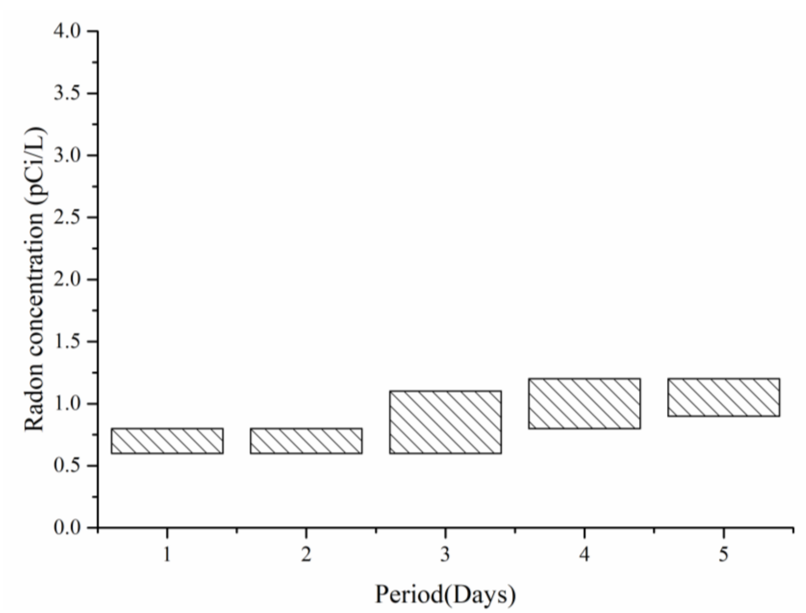

Fig. 3 **Radon concentration of MXene phase compound Ti_3C_2 for five consecutive days**

4. SUMMARY

A layered of titanium carbide Ti_3C_2 MXene was synthesized by pressureless sintering (PLS) method using a starting powder of TiH_2, Al and graphite for two hours. A layered of Ti_3AlC_2 was successfully exfoliated and fabricates a layer of 2D Ti_3C_2 by using an elegant exfoliation approaches. This is happening since the relatively weak bonding of Al layer compared to the Ti-C bonds. The layers of 2D Ti_3C_2 is quite thin between 0.1-1.0 μm. There are three phases was detected by XRD after the treatment which is Ti_3C_2 and the agglomerates of C and Al_2O_3. SEM image shows the agglomerates of this phase at the side of Ti_3C_2 layers. Radon concentration for this material for five consecutive days explains the radiation level of this material is under the suggestion value from US Environmental Protection Agency (EPA) which is 4 pCi/L.

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