

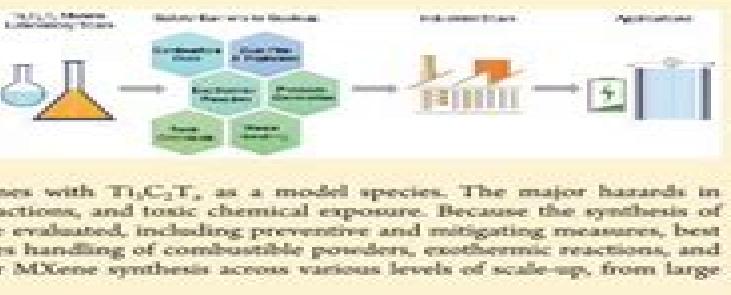
## Process Safety Analysis for $Ti_3C_2T_x$ MXene Synthesis and Processing

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### Supporting Information

**ABSTRACT:** MXenes are two-dimensional metal carbides with promising applications in energy storage and sensors. Guidelines for safe, scalable MXene synthesis are important due to ongoing efforts to scale-up production of these novel nanomaterials. Hazard identification before scale-up will allow for inherently safer designs prior to actual implementation. Here, we investigate hazards associated with MXene production, including MAX phase synthesis from raw materials, etching of the MAX phase to MXene clay, exfoliation to MXene nanosheets, and postprocessing of MXenes with  $Ti_3C_2T_x$  as a model species. The major hazards in MXene synthesis are the potential for dust ignition, runaway reactions, and toxic chemical exposure. Because the synthesis of MXenes is a multistep process, safety guidelines for each step are evaluated, including preventive and mitigating measures, best practices, and emergency procedures and responses. This includes handling of combustible powders, exothermic reactions, and harsh chemical etchants. This study is intended to facilitate safer MXene synthesis across various levels of scale-up, from large laboratory batches to commercial production.



### 1. INTRODUCTION

Nanomaterials are unique because they often present safety hazards in both synthesis and health. Over the last several decades, a number of studies on biological and health hazards of nanomaterials have been published, and these studies are crucial in understanding the effects of nanomaterials on humans and the environment.<sup>1–3</sup> Despite these recent advances, there is a stark absence of research assessing the process safety of nanomaterials synthesis and scale-up. The number of novel nanomaterials synthesized by researchers and small-scale manufacturing companies is increasing; thus, an intimate understanding of the process safety implications during laboratory synthesis and its scale-up is vital for faster and safer implementation of nanomaterials. A number of process safety incidents have occurred during scale-up, including several incidents in laboratories because of a lack of proper hazard identification.<sup>4</sup> For example, in 2010, a graduate student was injured when trying to scale-up a conventional process involving hydrazine perchlorate derivatives synthesis from 300 mg to 10 g.<sup>5</sup>

One of the primary causes of safety incidents in laboratories is a lack of knowledge of safety issues arising during the synthesis route; this is especially true in cases of new materials synthesis and novel processes where the hazards of the reactions are yet to be discovered. While most universities and research laboratories rightfully require researchers to conduct a safety analysis to identify and address potential hazards, these analyses cannot be thorough if the crucial information such as heat and gas generation rate by reaction, byproduct formation, thermal stability, etc. are lacking. This crucial information is

not immediately known for novel process development in the laboratory. In addition, the guidance for completing a thorough hazard assessment is often unavailable as the scientific community only recently adopted the concepts of process safety by the means of loss prevention. As a result of these reasons, scaling up novel processes for nanomaterials synthesis can result in catastrophic incidents as hazards can compound if not done correctly. This work is intended to highlight the importance of preventive measures in order to reduce the number and severity of incidents associated with the synthesis and scale-up.

MXenes are a growing family of atomically layered two-dimensional metal carbides and nitrides with the chemical formula  $M_{1-x}X_xT_y$ , where M is an early transitional metal in the periodic table, X is carbon or nitrogen, and T is a combination of several terminal groups (i.e.,  $-F$ ,  $-OH$ ,  $-O$ ,  $-Cl$ , etc.). These materials are most frequently etched from  $M_{1-x}AX_x$  (MAX) phase powders, which contain post-transition groups 13–15 in the metal layer that are located between M and X layers in their crystal structure. Two-dimensional carbides can have tremendous applications as MXenes have impressive thermal and electrical conductivities. MXenes are currently being evaluated for use in lithium and nonlithium ion energy storage, fuel cell additives, supercapacitors, humidity and pH sensors, and lubricating additives.<sup>6–8</sup> To date, over 60 MAX

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# Synthesis Analysis Methods For Safety

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## **Synthesis Analysis Methods For Safety:**

**Synthesis and Analysis Methods for Safety and Reliability Studies** NATO Advanced Study Institute on Synthesis and Analysis,   **Synthesis and Analysis Methods for Safety and Reliability Studies** G. Apostolakis, S. Garribba, G. Volta, 2012-12-06 This book originates from the NATO Advanced Study Institute on Synthesis and Analysis Methods for Safety and Reliability Studies held at Sogesta Conference Centre Urbino Italy 3-14 July 1978. The Institute co-directed by Prof E J Henley and Dr G Volta was attended by 67 persons from twelve countries. The focus of the Institute was on theoretical and applied aspects of reliability and risk analysis methodologies. The Institute was composed of lectures, workshops and guided discussions. From the large quantity of written material that was used and produced during the Institute a number of papers introducing the most relevant research results and trends in the field have been selected. The papers have been edited partly rewritten and rearranged in order to obtain in the end an integrated exposition of methods and techniques for reliability analysis and computation of complex systems. The book is divided into four sections which correspond to fairly homogeneous areas from a methodological point of view. Each section is preceded by an introduction prepared by the Editors which aims at helping the readers to put in perspective and appreciate the contribution of each paper to the subject of the section.

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