



Chrysotile asbestos detoxification with a combined treatment of oxalic acid and silicates producing amorphous silica and biomaterial



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HIGHLIGHTS

- Oxalic acid leaching followed by TEOS addition leads to production of glushinskite.
- TEOS treatment of chrysotile with oxalic acid addition yielded to amorphous silica.
- Oxalic acid leaching followed by water glass encapsulated the asbestos fibers.
- Oxalic acid combined with water glass can be used in onsite asbestos detoxification.
- Transformation of chrysotile into an amorphous material was achieved.

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ABSTRACT

This study was primarily imposed by the ever increasing need for detoxification of asbestos and asbestos containing materials (ACM), with potential application onsite. The present work investigates potential detoxification of pure chrysotile (Chr) asbestos via a combined treatment of oxalic acid dihydrate (Oxac) ($\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$) with silicates, such as tetraethoxysilane (TEOS) (SiH_2O_4) and pure water glass (WG) (potassium silicate) (K_2SiO_3). These reagents used in the experimental procedure, do not cause adverse effects on the environment and are cost effective. The results of FTIR, XRD, optical and scanning microscopy coupled with EDS analyses indicated that all of the applied treatments destructed the Chr structure and yielded silica of amorphous phase and the biomaterial glushinskite from the Oxac reacted with brucite [$\text{Mg}(\text{OH})_2$] layer. Each of the proposed formulations can be applied for the detoxification of asbestos, according to priorities related to the specific products of the recovery treatment. Therefore, Oxac acid leaching followed by the TEOS addition is preferred in cases of glushinskite recovery; TEOS treatment of asbestos with subsequent Oxac addition produced amorphous silica production; finally Oxac acid leaching followed by WG encapsulated the asbestos fibers and can be used in cases of onsite asbestos and ACM detoxification.

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1. Introduction

The adverse environmental and health effects of asbestos are very well documented in literature [1]. In an attempt to eliminate the consequences of asbestos researchers have been directed to its treatment. Nowadays, there is no unifying legislation that specifies ACM and pure asbestos waste management. Therefore, the

most widely used method is deposition of ACM in landfills. Even when deposited in such ways, a pretreatment of the waste is necessary and possibly a long-term landfill restoration, too. Nonetheless, landfilling is not the proper method because of the waste still remains hazardous. Instead of this, an environmentally sound and cost effective detoxification method should be developed, with a future option of an onsite application. Last decades onsite detoxification treatments of chrysotile asbestos gain significant ground because they provide low risk and cost efficiency. These treatments include encapsulation of chrysotile fibers in a matrix of either cement or resin [2,3] as well as insulation of asbestos by using spray foams [2]. Chemical treatment constitutes an alternative in-situ process. According to recent studies usage of oxalic acid

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Table 1
Chemical composition of chrysotile asbestos.

Main oxides	Percentage
MgO	38.46%
SiO ₂	41.39%
Fe ₂ O ₃	5.47%
CaO	0.43%
Al ₂ O ₃	0.24%
Loss on ignition	14%

under ultrasonication fully degrades chrysotile asbestos fibers [4], eliminating health and safety risks. Furthermore, thermal treatment of chrysotile is also recommended. Through this method, chrysotile fibers are entirely transformed to forsterite which is a non-hazardous material. However, this method has a major drawback which is related to the consumption of energy which is higher than the above mentioned methods [5]. After 2006, relevant regulations became even stricter, reducing the permissible levels of asbestos-containing products to 0.1 wt%. Consequently, any asbestos mining and/or manufacturing process have been since minimized and any exposure to it would inevitably result from renovations or demolition wastes [6]. Moving on to this direction, researchers have investigated and established various detoxification methods, such as detoxification of ACW prior to their disposal or even their recycling. Contrary to the legislation, there are, still, some activate asbestos mines. In 2011, 2.03 million tons of Chr were excavated mainly in Russia, China, Brazil, Kazakhstan and Canada [7].

The structure of Chr (Table 1) is characterized by a continuous repetition of Mg(OH)₂ and acidic SiO₂. According to its structure, Mg-octahedral sheet is bonded to Si-tetrahedral via O-atoms [8]. Besides its strict structure, strong acids such as HCl, H₂SO₄, and HF [9–11] have already been used to destroy the external sheet of brucite. However, these acids are harmful to human health and should be avoided [12]. Furthermore, an experiment of combination of strong acids and fluoride ion source resulted to completely dissolution of magnesium oxide and silica of Chr [8]. For the conversion of asbestos to a non-toxic material under acidic conditions, the pH of solutions should ideally range from 0.6 to 2. However, these limits could be deviated since the results of the present study have demonstrated that detoxification of Chr can be achieved in both acidic and alkali conditions.

An earlier research has reported the use of sulfuric acid with a direct attack on asbestos to convert the latter into a non-toxic material [13]. This research revealed the decomposition of asbestos when reacting with acids, such as sulphuric acid or in the aqueous solution of ammonium sulfate. Asbestos is usually treated by heating, mixing, cutting and stirring in acid conditions. Although, such strong acids can be used to detoxify the asbestos fibers, they are still considered even more hazardous than Chr asbestos [12]. On the other hand, weak organic acids, such as acetic and lactic acid, have also been used to convert asbestos-containing materials into non-toxic [14]. Rozalen and Huertas [13] investigated the mechanism of Chr modification using different acids at pH 1. Furthermore, the use of polycarboxylic acids, such as Oxac, is considered advantageous due to its capability of converting asbestos into non-toxic materials, with or without adding alkali [15]. According to literature data, mixing Chr in an Oxac solution has resulted in the production of magnesium oxalate dihydrate, magnesium hydroxide (brucite) and amorphous silica [13]. The Oxac leaching of Chr can be achieved by a reaction between brucite and Oxac. As a result, the magnesium hydroxide of brucite sheet is decomposed. Oxac is a moderate organic acid, in terms of acidity and toxicity. It is a cost efficient choice of a reagent since it is a byproduct of chemical industry. Additionally, it is found in soils and lichens. Therefore, in accordance to environmental requirements both reuse of waste and

cost effective detoxification treatment were achieved. It has been demonstrated in literature that when lichens contact with Chr, the detoxification of fibers is achieved [16,17].

In the present study, in order to accomplish the detoxification of Chr into a non-hazardous material, a weak acid treatment using Oxac combined with reagents promoted a silylation process, such as pure water glass (WG) and TEOS, has been investigated. The Oxac-leaching can transform the natural polymer of Chr, by removing the Mg sheet, to an excellent source of SiO₂. The silylation of Chr is a simple process and once implemented correctly, a silicon polymer is obtained [18]. Its properties are enhanced compared to the corresponding synthetic silica gel [18,19]. The addition of TEOS in an acid solution of Oxac leads, through the silylation process, to a sol–gel material. The WG addition in the same solution leads to the encapsulation and transformation of fibers into a resinous coating. Therefore, in this combined treatment method, where the Oxac assisted in the transformation of fibers, which were subsequently embedded into the silica network created either by TEOS or pure WG through the silylation process. Furthermore, WG creates a type of vitrification on the top of the material which prevents the diffusion of the fibers.

2. Materials and methods

2.1. Materials

Distilled H₂O was used in all the experiments so as to facilitate the desired hydrolysis process of TEOS and WG. The reagents TEOS, water glass (WG), isopropanol (ISP), oxalic acid dihydrate (Oxac), were purchased by Sigma–Aldrich (Germany).

2.2. Synthesis

As it has already been stated above, a successful silylation processes, with the appropriate reagents, leads to a silicone polymer, which is soluble in various organic solvents. Therefore, the silylation process is a possible treatment that leads not only to a safe disposal of Chr, but also to its recovery as a non-toxic and valuable material [19]. In addition, the produced polymer has a silicone resin type, possibly attributed to the original silica structure of Chr [18]. The environmentally friendly TEOS and WG, in conjunction with Oxac, were chosen in order to achieve the transformation of Chr to a non-toxic, new sol–gel.

The mixing of Chr with water does not affect its properties. For comparison purposes, therefore, a mixture of Chr with distilled water was used as a reference sample Ref. [1]. This sample was prepared and placed on a shaker, where it remained during the whole experimental procedure and was further examined using FT-IR, XRD and SEM.

2.2.1. Treatment with oxalic acid–TEOS (AOxT)

AOxT treatment consisted of the dispersion of Chr in a solution of Oxac first, and then the addition of a solution of TEOS in ISP, as listed in Table 2. The molar ratio was Chr/Oxac/TEOS = 1/9/124. The pH value of AOxT was measured equal to 1.1.

2.2.2. Treatment with TEOS–oxalic acid (ATOx)

ATOx treatment differs from AOxT in the sequence of the raw materials during the detoxification process. In the case of ATOx, Chr was first dispersed in TEOS with ISP and after 5 days Oxac was added into the dispersion. The pH value of AOxT was measured equal to 2.6.

2.2.3. Treatment with oxalic acid–pure water glass (AOxWG)

AOxWG treatment involved first the dispersion of Chr in Oxac and then the addition of WG. In AOxWG treatment the pro-

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