# A TECHNICAL APPROACH TO INERTIZE SERPENTINE TAILINGS THROUGH AMORPHIZATION OF CHRYSOTILE

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## Abstract

Chrysotile asbestos mine operations over several decades has generated significant amounts of tailings. Due to the adverse health effects associated with exposure to chrysotile-fibers, some countries have banned its use, which has led to the closure of mines. These residues may have an economic value, but the presence of residual fibres impedes on their direct recycling. In this article, the combination of mechanochemical and thermal treatments is proposed to make the material inert and available for several applications at an affordable cost by maximizing energy consumption to destroy the fibers. The technical approach could be applied to other asbestos mining residues.

Keywords: asbestos, chrysotile, serpentine, mechanochemical, amorphization, dehydroxylation, inertization, circular economy

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## 1. Introduction

Canada has been one of the largest asbestos chrysotile producers in the world. Most of the mining operations have taken place in the province of Quebec where chrysotile was mainly mined in open pits from serpentine ore [1,2]. The fibre recovery processes generated two categories of wastes: the waste rock mostly composed of coarse-grained fragments with highly variable granulometry, and the tailings consisting of residue directly produced from ore milling and chrysotile extraction. This is the second type that raises commercial interest. Serpentine, brucite and magnetite are major constituents of tailings, and a significant proportion of forsterite can also be identified. Trace elements such as nickel, chromium and cobalt are additionally present [2-4]. Similar residues are found in other countries where chrysotile has been or is still exploited, such as the United States and Russia [1,5,6].

From a nomenclatural standpoint, *Asbestos* is a generic name used to designate naturally-occurring fibrous silicate minerals. Based on their physical and chemical properties, there are two major families of asbestos: amphibole with five subtypes (crocidolite, amosite, anthophyllite, actinolite, and tremolite); and serpentine with only one subtype (chrysotile). The latter was and remains the only type of asbestos mined on a large scale [6-8]. The three most important polymorphs of serpentine are lizardite, antigorite and chrysotile which are expressed as $Mg_3Si_2O_5(OH)_4$. Lizardite has an ideal layer topology which forms a platy morphology. Antigorite is modulated and shows a corrugated structure. The basic crystal structure of chrysotile is a cylindrical lattice and makes up the most common form of asbestos. Serpentine has a layered structure with one layer consisting of linked silicate tetrahedra joined to a brucite octahedral layer [2,8]. Chrysotile occurs most commonly in the mineral under cross-fiber vein shapes. Chrysotile-fibers are very flexible with a

high tensile strength and resist corrosion, fire and sometimes acids, features that have strongly advocated its use [1,2,7].

Crystal-chemical transformation induced by thermal treatment of chrysotile is the most common process used to destroy its fibrous structure, which is unstable at high temperatures. Thermal decomposition is a dehydroxylation reaction where structurally bound hydroxyl groups –OH are removed from the solid leading to the formation of new mineral phases. This reaction generally occurs above 500°C according to the origin of chrysotile (in pure state or in a mineral), particle size, atmospheric conditions of calcination and the type of treatment that normally entails either isothermal or prograde heating. The dehydroxylation process also depends on the thickness of the layer if calcination is conducted in a stationary bed. The chrysotile decomposition sequence is quite complex and proceeds via the formation of intermediate phases in conformity with its topotactic nature. In brief, chrysotile is transformed into forsterite $Mg_2SiO_4$, associated with enstatite $MgSiO_3$ as the temperature increased [9-11].

Calcining large quantities of serpentine tailings with residual chrysotile-fibers[1] would require a lot of energy and inevitably leading to expensive processing costs. Since residues should be first ground in whole or in part, selection of grinding techniques that affect the crystal structure of serpentine polymorphs, making it more suitable for calcination is proposed. The precursor mechanical process will induce a reduction in the thermal energy required to produce a harmless material, thus a more economical way of doing things. To that end, this article presents at first the results of some previous work on mechanochemical and thermal transformation of hydrous magnesium silicates as a basis, followed by a description of the proposed technical approach.

## 2. Mechanochemical treatment

We know that size-reduction processes cause on both the crystal and chemical changes of inorganic solids: mechanical activation and modifications in surface properties; polymorphic transition and alterations in physical properties of the bulk phase; decrystallization and amorphization; mechanochemical solid-state reactions. Among others, the changes allow easier thermal decomposition, and greater availability to react or intensify dissolution of a leaching process [12,13].

In that sense, Zhang et al. [14] has shown that serpentine is progressively transformed from a crystalline state into an amorphous one by dry grinding in the same way as a heating process. The sample used for the study was first crushed by a stamp mill and then grinded by using a planetary ball mill. The experimental results suggest that the amorphization of serpentine is attributed to the disordering around the magnesium local structure by mechanical treatment. According to thermogravimetric curves, the dehydration of ground materials is observed to occur at considerably lower temperatures than that for the starting sample. Furthermore, the weight loss of the samples increases as the grinding progresses and seems to be attributable to the release of the –OH base group. The authors also deduced that the local structure of silicon-oxygen bonding Si-O– is disordered to some extent by a prolonged grinding time. The amorphization of serpentine improves extraction of both magnesium and silicon at room temperature by acid solutions (1N HCl and 1N

[1] For example, in the Thetford region of Quebec, chrysotile content in tailings is variable and can reach 40% (v/v) due to extraction methods that have improved over time [2,4].

$H_2SO_4$)[2] where magnesium is preferentially extracted, and to a greater extent, over a short sample grinding period. Additionally, it was observed that the size reduction of the sample predominates in the beginning stage of grinding while increasing the specific surface area (SSA) and that inversely, the fine particles aggregate upon prolonged grinding with a diminution of SSA.

Likewise, Plescia et al. [13] demonstrated that asbestos-containing waste, as well as chrysotile or pure amphibole, may be transformed in amorphous form with a complete modification of its fibrous morphology under the effect of an aggressive mechanical impact. The particles found on all samples after grinding treatment had sizes varying from about 10–50 μm which the larger particles are mainly formed of aggregates, a finding similar to Zhang's. A vibratory disc mill was used to simulate the operating conditions of a mechanochemical reactor.

Another study carried out by Bloise et al. [15] investigated the effects that dry grinding in an eccentric vibration mill has on tiny mineral fibre samples, such as chrysotile, amosite and crocidolite. Mechanical treatment was accomplished in short periods of time, i.e. 10 minutes or less. Experimentation showed that grinding causes a decrease in crystallinity, the formation of lattice defects and size reduction with progressive formation of agglomerates in the samples. A decrease of the dehydroxylation enthalpy as grinding time increased was also observed, signifying the possibility to destroy the fibres at lower temperatures than those used for the destruction of the mechanically untreated fibres. In other words, fibres with structural defects or partially destroyed require less thermal energy to be transformed into non-hazardous material. The results showed that partial dehydroxylation already occurred during the grinding process.

The transformation of a solid material is highly dependent on the device and the operation conditions. Some processes are more easily scalable than others. Grinding equipment used at the laboratory scale in the studies cited involves different combinations of stresses that reduce the size of the grains or fibres and weaken the structure of solids. According to photographic analysis, the surging phenomenon of the motion of balls in the planetary mill predicts that the grinding mechanism consists of compressive, abrasive and shear stress [16]. Planetary ball mills are well known and used on laboratory and pilot plant but the great expense of scaling them up to full production size has so far limited its widespread use in manufacturing, although available [17].

About the eccentric vibratory mill, it is known that it performs in the field of mechanochemical activation and micro-grinding. Unlike conventional vibratory mills with homogeneous circular vibrations, this design version causes elliptical, circular, and linear vibrations. Due to a major increase in the acceleration of the individual grinding media, the throughput is increased. The compression and the shear stress are the main forces acting on the matter. Eccentric vibratory mills can be operated in batch or continuous mode at industrial scale while the conventional model has a limited scalability [18].

Comminution in stirred media mills[3] is a frequently applied method towards the production of micron- and submicron particles. This type of mill is also appropriate for mechanical activation due to the high energy density which is higher than other high energy density mills like planetary ball mills or vibratory mills. The main stresses are compression, shear, and impact. It is often used for

[2] Normality hydrochloric acid and sulfuric acid.
[3] Also called attritor mills.

fibrous materials. Attritor mills scale up easily and efficiently to large production machines and can run in batch or in continuous mode under heating to aid in the grinding process [19,20].

## 3. Thermal treatment

It is important to note that thermal transformations determined from prograde heating and isothermal heating are different due to the inverse relationship between decomposition temperature and heating duration. It has been shown that thermal events are typically observed at higher temperatures in prograde heating experiments, whilst these events occur at a much lower temperature in isothermal mode. Ramping up the temperature may serve to comparing different serpentine minerals or the particle variation size of a sample by contrasting their thermal analysis results, but it should not be used to establish temperatures of thermal stability [9-11].

Weber et al. [21] demonstrated in a stepwise heating study that lizardite, antigorite and chrysotile have their own decomposition temperature and heat of dehydration, as well as a serpentine ore containing a mixture of these polymorphs. Further studies showed that chrysotile, or more broadly constituents of a serpentine sample, undergo a mineralogical change when heated depending on the length of heating time at a given temperature [3,11,22]. As Crummett's work shows [11], pure chrysotile was no longer present above 575°C and 24 hours whereas chrysotile heated to 800°C survives for only minutes.

In a research project of Naumann et al. [23], it was found that microporosity of a sample has a pronounced influence on chrysotile dehydroxylation by the action of heat, such by the ease with which molecular water can be removed from reaction sites. Specimens of high surface area, in their natural state or induced by grinding and/or chemical treatment, indicate an open texture dehydroxylate to form an *x*-ray-amorphous phase that remains stable for a while under an increase in temperature after the dehydroxylation process has taken place. For them, forsterite formation occurs at higher temperature and in a narrow range. In other words, the recrystallisation of chrysotile from topotactic structure to more rigid structure is postponed. Similar results were obtained by Brindley et al. [24] with serpentine samples under isothermal heating conditions in air. The rate of recrystallization to forsterite is shown to have an inverse relationship to the rate of dehydration. This linkage was established by experiments in which coarse serpentine was found to dehydrate more slowly and recrystallize more rapidly than a finely ground portion of the same material. Analogous behavior was observed for massive specimens: surface layers dehydroxylated rapidly to form an amorphous phase, while the core of matter dehydrated more slowly and transformed readily to forsterite. This result is interpreted in terms of the damage inflicted on the crystal structure of serpentine by the dehydration reaction: open-textured materials allow water diffusion easily.

Publications present the controlled calcination of hydrous magnesium silicates of the serpentine group as a method for producing active magnesia MgO. The degree of activation is measured by the solubility of magnesium in weak acids, for example carbon dioxide $CO_2$ solution. The authors emphasize that the amount of residual water combined with mineral has an impact on activation and that maximum occurs at around a third of the initial content. Residual water refers to the amount still retained by the calcine and which when fully ejected will result in a dead burned calcine, i.e. a calcine incapable of showing further loss on ignition. [3,22]. In the same vein, thermal activation of serpentine improving its carbon sequestration reactions has been known for some time. Evidence

of studies suggests that structural disorder of the matter plays a dominant role in carbonation and that the return to a structure in the form of forsterite and/or enstatite must be prevented by careful consideration of the treatment temperature to avoid over heating. $CO_2$ capture and storage through the use of magnesium-rich minerals are a mitigation strategy to limit global warming [25,26].

With regard to the atmosphere in which the heating process takes place, the study of Naumann et al. [23] mentioned above demonstrated that environment conditions influence the dehydroxylation process of chrysotile: the use of dry nitrogen $N_2$ purge gas instead of wet $N_2$ or air lowers the hydroxyl release temperature.

Several types of furnaces can be used on an industrial scale to transform serpentine tailings, with the basic criterion being the temperature to be reached, residence time followed by gas flow: tunnel oven, rotary kiln, fluidized-bed system.

## 4. Discussion about the technical approach

### 4.1 Context

The chrysotile industry has generated huge quantities of residues whose composition opens the door to broad application prospects. In this sense, the proposed inertization process aims to transform them into a safe and versatile product on stockpiling sites while reducing the existing environmental liabilities on affected communities. Currently, chrysotile, being a hazardous substance under the Globally Harmonized System of Classification and Labelling of Chemicals (GHS), is subject to Canada's health and safety rules as well as Transportation of Dangerous Goods (TDG) regulations and similar standards in other countries. This framework, while necessary, limits the transport and the use of these residues. At a time when the circular economy is being advocated, the technical solution consists of altering the chrysotile-fibers they contain through a comminution processing, which requires less energy to complete its inertization[4] by heat treatment, thus producing a marketable material.

According to the literature consulted, the forces at play in mechanochemistry have a significant impact on lattice defects of serpentine polymorphs or amphibole species, even a partial dehydroxylation. The stresses applied to the material depend on the mill selected. The examples cited show the results of grinding materials that were finely ground during experimentation. Although grinding to a fairly fine mesh (–44 µm) could increase the yield of structural changes, from an economic point of view, it is not advisable unless the subsequent application of the product requires a smaller grain size. Moreover, fine grinding may cause particle agglomeration, which is undesirable. Researchers have established that at the initial comminution stage the size of the material is reduced, while at the same time its specific surface area increases. Next comes an agglomeration phase after a certain maximum fineness is achieved over time, reducing the surface and lowering process energy efficiency [19,27]. Aggregates formation have to be avoided to preserve high surface area of the matter to pursue its transformation more easily and with less energy in the next heating stage: structural defects favor the diffusion of –OH and the formation of an amorphous phase as stated in the previous section. Wet grinding is not recommended because

[4] Inertization means that all chrysotile is in a non-hazardous form by a complete destruction of its fibrous morphologie.

chemical reactions of the stressed particles with the liquid phase may occur, then possibly affect the dehydroxylation process.

Since thermal transformation of minerals depends on temperature and duration of treatment, relatively low temperatures are not advocated, for the reason that the time required would be too long to be within economical limits. In addition, it is preferable that temperature and processing time promote the amorphous state of serpentine polymorphs, limiting the formation of forsterite. This condition will lead to more commercial opportunities for the product by allowing direct use or subsequent transformation. The atmospheric conditions under which processing takes place have an impact too: the use of a dry and inert purge gas with sufficient flow to evacuate rapidly the vapour released has proved beneficial. The presence of moisture or oxygen in the environment may engender the appearance of a backward reaction which makes the process slow down and requires more energy.

Milling and heating apparatus have been commented on and/or proposed previously as an aid to selection, but without limitation. The following subsection presents the main stages of the transformation process, which can be adapted to the material to be inertized.

### 4.2 Inertization process

Under safe handling conditions on the storage site, then under secure production environment, the inertization process (Figure 1) could begin by a coarse grinding stage with a heating option of low-moisture or dried up residue according to grain size. Over a fairly long mining period, serpentine ore was pre-dried before the fiber was extracted, in order to meet separation standards under anhydrous conditions. The raw fiber was separated from the rock by using a series of impact comminutions with minimal incidence on fiber qualities, followed by vacuum screening [1,2,5]. Some of the residues are therefore already relatively dry. The objective is to fragment the matter by a grinding technique which also noticeably initiates structural alteration of chrysotile. Careful choice of equipment will have energy-saving repercussions on the subsequent grinding step where the process begun will continue more intensively.

After that or initially, the residue might be subjected to dry magnetic separation to remove the iron in the form of magnetite. Concomitantly, Martinez's tests [28] have shown that chrysotile particles contained in serpentine ores were found to respond to a magnetic field depending on the intensity of the field in relation to the size of the ore particle. Chrysotile with partial structural defects was however not discussed. By this optional route, a magnetite-chrysotile concentrate would be obtained, with a so-called *non-magnetic fraction* containing leftover fibers. If native nickel-iron known as awaruite $Ni_2Fe$ to $Ni_3Fe$ is present in the residue[5], attention could also be paid to the impact of the applied field on it. A recent study conducted by Seiler et al. [29] has demonstrated the magnetic properties of this mineral.

Crude serpentine residue, finer altered residue or fractions from magnetic separation are then subjected to mechanochemical treatment. The choice of fine grinding technology and operating conditions must promote deterioration of the chrysotile structure as much as possible while taking

[5] For information, native nickel-iron has been found over a wide area in the serpentinized asbestos bearing rock of the Eastern Townships of Quebec [30].

into account the reduction in particle of the submitted matter. The shape of lizardite and antigorite will be damaged too but the target polymorph is chrysotile since it is the compound to be inertized. Moreover, in Plescia's reporting [13], it is stated that magnetite $\alpha$-$Fe_3O_4$ tends to transform into maghemite $\gamma$-$Fe_2O_3$, and then into hematite $\alpha$-$Fe_2O_3$ when mechanically treated. More than one operative protocol has to be set depending on the feed.

The variation of determinant structural parameters, such as specific surface area, crystallinity/amorphism, emergence of new compounds and particle size, should be measured as a function of injected milling energy input over a period of time.

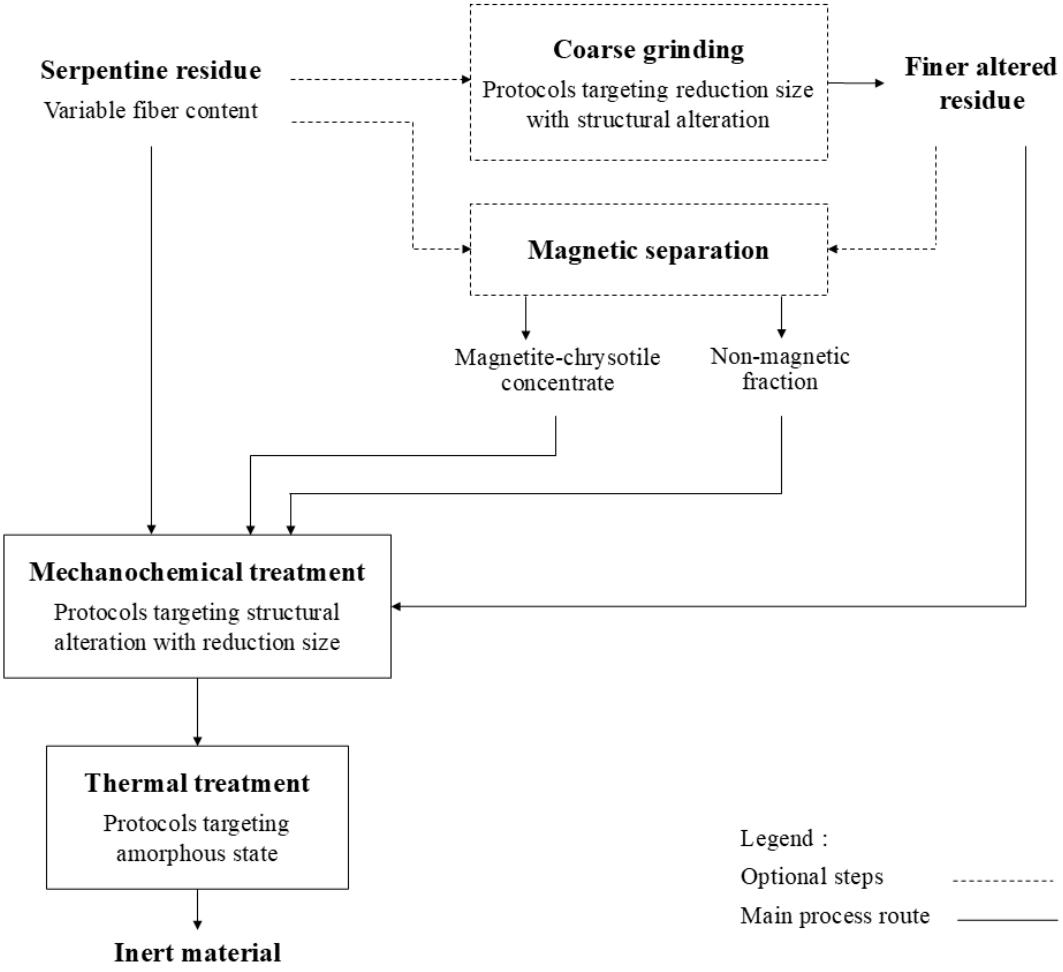


Figure 1: Process block-diagram

Fine materials are then subjected to heat treatment to finalize the conversion of chrysotile into a harmless substance via the destruction of its fibrous structure. In conjunction, thermal process must retain as much matter as possible in amorphous state: the effect of temperature and time applied on

a mixture of minerals[6] with different levels of lattice defects and dehydroxylation of its serpentine polymorphs obtained by grinding is specific to it; these structural changes will contribute to delay the forsterite formation and vice versa will preserve the amorphous state. In sum, the optimum temperature-time combination has to be defined for each type of material on the basis of its composition and structural disorder of lizardite, antigorite and chrysotile, according to studies cited above.

Samples analysis by x-ray diffraction combined with Rietveld refinement could be a method to identify and quantify the crystalline phases of a mixture, as well as its amorphous content. In addition, the use of several analytical techniques to accurately detect chrysotile, and more broadly all fibrous structures, is essential.

### 4.3 Product uses

Including but without limitation, the product with amorphous content can be used as is in various applications or after further processing:

- Agriculture sector. As an additional source of magnesium and an amendment to increase the soil's pH by the availability of magnesia; calcined with apatite or phosphorite to make calcium magnesium phosphate fertilizer.
- Cement industry. When ground to a cement fineness, it will act as a hydraulic binder due to its amorphous nature, which reacts with water to form a new magnesium silicate hydrate.
- $CO_2$ capture and storage. Containing high levels of magnesium which can be converted into carbonate form where structural disorder of the matter constitutes a dominant factor controlling the availability of magnesium in the carbonation efficiencies[7].
- Production of salts, oxides, or metals. Magnesium, chromium, cobalt and nickel, which are on Canada's list of critical minerals and essential for a green digital economy, can be easily extracted by acidic or basic wet process and generate amorphous silica in parallel.
- Heat storage or refractory products. The iron-rich product of magnetic separation might be useful for the preparation of heat storage cores and refractory bricks.

## 5. Conclusion

The technical approach adheres to circular economic strategies, in particular via: the recycling of mining residues through a transformation process with a view to reintroducing them into new production cycles while preserving untapped mineral resources; the reducing energy consumption to inertize residues, thus protecting the ecosystems that generate it; a collaborative economy by making a multi-purpose product to intensify its use.

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[6] Serpentine, brucite, iron oxides, etc.

[7] Consideration: the carbonate matter obtained has a restricted use since the stored carbon must remain there to conserve a positive contribution to the emission balance, for example as backfill material. Requires characterization to assess environmental impact.

To implement the solution, tests must be carried out and samples subjected to several analyses to determine the conditions under which a serpentine residue should be mechanochemically treated then heated in order to yield a maximum quantity of amorphous matter while ensuring that chrysotile is undetected. This design strategy makes easier to sell the product in different business sectors. More broadly, the inertization approach could be used to treat residues containing other asbestos compounds. Faced with such recovery potential, the game is worth the candle.

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