

Characterization and classification of unregulated mineral fibers: The case of fibrous datolite from Rio Manubiola Valley, Parma, Italy

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ABSTRACT

The widespread presence of mineral fibers necessitates their thorough identification, characterization, and assessment of potential health effects. This comprehensive approach is crucial for assessing and controlling exposure to hazardous fibers such as those in asbestos minerals. Within this framework, we present the first detailed investigation of fibrous datolite $\text{CaBSiO}_4(\text{OH})$, a calcium boron hydroxide nesosilicate. Although this mineral commonly crystallizes in non-fibrous habits, a rare pearly-white fibrous deposit was identified within an opicalcite from the Rio Manubiola Valley (Parma, Italy). While datolite is an industrial boron ore, its fibrous habit and potential human toxicity are currently unregulated. Therefore, alongside the crystal chemistry of the sample, this work includes an evaluation of its potential human toxicity and carcinogenicity by applying the Fiber Potential Toxicity Index (FPTI), a quantitative predictive model based on the physical-chemical and morphological parameters of a mineral fiber.

Fibrous datolite is both chemically and structurally similar to its non-fibrous counterpart [space group $P2_1/c$, $a = 4.83679(1) \text{ \AA}$, $b = 7.61346(2) \text{ \AA}$, $c = 9.63754(3) \text{ \AA}$, $\beta = 90.156^\circ$]. Regarding morphological parameters, the average length, width, and aspect ratio of individual datolite fibers are $9.80 \mu\text{m}$ (s.d. 5.39), $1.32 \mu\text{m}$ (s.d. 0.82), 9.53 (s.d. 6.05), respectively, classifying them respirable according to the World Health Organization (1997) fiber size criteria [length $\geq 5 \mu\text{m}$, width $\leq 3 \mu\text{m}$, and aspect ratio (length/width) $\geq 3:1$]. Nevertheless, based on the FPTI model, fibrous datolite demonstrates one of the lowest potential toxicity/carcinogenicity scores, specifically $1.50(0.1)$, compared to all previously evaluated mineral fibers. This exceptionally low score is primarily attributed to its lack of biodurability and absence of iron or other metals in the crystal structure.

Given its predicted null toxicological potential, fibrous datolite can be proposed as a potential negative standard for in vitro cell toxicity studies, aligning with the use of other non-toxic fibers like wollastonite.

Keywords: Mineral fiber, datolite, crystal structure, FPTI, toxicity

INTRODUCTION

In the Introduction of their famous book *Asbestos and Other Fibrous Materials* (Skinner et al. 1988), the authors state that “Fibers are everywhere around us.” At least 388 mineral species display a fibrous crystal habit [see Appendix 1 in Skinner et al. (1988)]. These include native elements, sulfides and sulfo-salts, oxides, hydroxides, halides, carbonates, nitrates/iodates, borates, sulfates, selenates/tellurates/chromates, phosphates, arsenates, vanadates/vanadium-oxysalts, tungstates/molybdates, organic, silicates, and aluminosilicates. This list is not exhaustive; it is expected that the number of mineral species displaying a fibrous (and eventually asbestiform) crystal habit is likely much greater

than 388. For example, the list does not include the borosilicate species datolite, described in this work.

The potential harm to human health from inhaled biodurable fibrous minerals is widely acknowledged. Specifically, the silicate “asbestos” species and the aluminosilicate (zeolite) erionite are considered as the most hazardous (Wylie 2017), both classified as “carcinogenic to humans (Group 1)” by the International Agency for Research on Cancer (IARC) (IARC 2012). Fluorooedenite represents another mineral fiber identified as a human carcinogen (Gianfagna et al. 2003; Cardile et al. 2007; Andreozzi et al. 2009; Fantauzzi et al. 2012). Considering the vast number of mineral species that exhibit a fibrous habit, the possibility that others, beyond those currently recognized, could present health risks cannot be discounted. Therefore, advancing knowledge and research in this domain is of paramount importance. The overarching goal is to identify all fibrous minerals capable of

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producing asbestos-like health effects, thereby warranting their inclusion in exposure assessment and control strategies. Moreover, the determination of maximum safe exposure levels for these minerals necessitates rigorously conducted studies (Carbone et al. 2016).

Datolite, $\text{CaBSiO}_4(\text{OH})$, is a calcium boron hydroxide nesosilicate, often formed during low-temperature hydrothermal events and low-grade metamorphic processes. Datolite can also crystallize in skarns, some regionally metamorphosed rocks, and during diagenesis of evaporites and other sediments. The most familiar occurrence of datolite is in secondary veins and cavities associated with calcite, prehnite, and zeolites in basic hypabyssal and volcanic rocks and hydrothermal deposits (Grew 1996). The scientific interest in datolite mainly lies in its potential use as a geochemical marker (Bellatreccia et al. 2006; Zaccarini et al. 2008). Aesthetically appealing crystals of datolite are appreciated by mineral collectors and gemmologists (Graham et al. 2018; Marinoni et al. 2023). From an industrial perspective, datolite is exploited as boron ore for the glass and ceramic industries thanks to its abundance in some skarn deposits, such as the giant deposits of Dal'negorsk, Russia (Grew 1996; Baskina et al. 2009; Kiss et al. 2012).

From a crystal-chemical viewpoint, datolite is generally close to its ideal formula $\text{CaB}(\text{SiO}_4)(\text{OH})$. For example, recent studies of several datolite samples from ophiolitic basalts of the Northern Italian Apennines confirmed the almost complete absence of trace elements and site substitutions (Zaccarini et al. 2008; Rinaldi et al. 2010). Only a few studies (Grew 1996) report impurities of Al_2O_3 , FeO , and MnO approaching 1 wt%.

The crystal structure of datolite was first determined by Ito and Mori (1953), and shows a layered structure (Online Materials¹ Fig. S1) with the regular repetition along the [100] direction of two different layers. The first layer consists of alternating SiO_4 and $\text{BO}_3(\text{OH})$ corner-sharing tetrahedra, forming 8- and 4-membered rings (Online Materials¹ Fig. S1a). The second layer is composed of 8-coordinated Ca polyhedra arranged in 6-membered rings (Online Materials¹ Fig. S1b). The crystal structure was later revised by many authors focusing on structural details, similarities with other calcium boron hydroxide silicates, or non-ambient crystal structure (Rinaldi et al. 2010 and literature therein).

The typical morphologies of datolite described in the early literature (Grill 1928; Bertolani 1948) are reported in Online Materials¹ Figures S2 and S3. To the best of the authors' knowledge, only two subsequent reports mention fibrous datolite. Symes (1977) documented fibrous datolite from the Merehead Quarry (Cranmore, Somerset, U.K.) describing the mineral as "fibrous radiating masses of datolite" and "thin crust, up to 3 mm thick, of milky white fibrous datolite." Sonnet (1981), in his description of the mineral assemblage associated with the new mineral burtite from El Hamman (Central Morocco), reported "Under the microscope, this datolite replaces wollastonite, as shown by remnants of a fibrous cleavage sometimes preserved in the datolite ... In places, the minerals are partly replaced by chalcedony and late pearly fibrous datolite are totally dissolved, with the creation of vugs". Synthetic fibrous (and in some cases asbestiform) borosilicates were also obtained together with datolite from the $\text{CaO-B}_2\text{O}_3\text{-GeO}_2\text{-H}_2\text{O}$ system at

415 °C; however, their crystallographic description and identification remained partial (Barrer and Freund 1974).

This study originates from the rare finding of a pearly-white fibrous datolite in an ophicalcite of the Rio Manubiola Valley (Parma, Italy), sometimes also accompanied by fibrous pectolite. This area is known in the Italian literature for the occurrence of several mineral species (Adorni 1993). Notably, exceptionally large datolite crystals (up to 10 cm) of high quality were found in the Valmozzola area about 50 years ago (Bartoli et al. 2008). To date, this is the first investigation of fibrous datolite from this area, and it contributes to the very scarce existing literature on fibrous datolite in general.

Integrating crystal chemistry with an assessment of potential human toxicity and carcinogenicity, this investigation aimed to identify fibrous minerals possibly exhibiting health risks comparable to asbestos and erionite. The Fiber Potential Toxicity Index (FPTI), a quantitative predictive model based on the physico-chemical and morphological parameters of mineral fibers (Gualtieri et al. 2021), was used to assess this potential risk. The model evaluates the morphometric, chemical, biodurability-related, and surface parameters of mineral fibers responsible for adverse effects in vitro and in vivo (Di Giuseppe et al. 2021a; Gualtieri 2021, 2023). The FPTI model considers the morphometric parameters (mean fiber length and diameter, crystal habit and curvature, density, hydrophobic character, specific surface area), chemical parameters (iron content, content of ferrous iron, surface iron and its nuclearity, content of metals other than iron), biodurability related parameters (dissolution rate, rate of iron dissolution/release, rate of silica dissolution/release, rate of release of metals from the fiber), and surface reactivity (zeta potential, aggregation state of the fibers in suspension, cation exchange). A detailed description of each parameter of the model is provided in Gualtieri (2018). In the last years, the FPTI model has been used to evaluate numerous mineral fibers like asbestos (Gualtieri 2018), fibrous glaucophane (Di Giuseppe et al. 2019), wollastonite (Di Giuseppe et al. 2021b), erionite (Patel et al. 2024), and fibrous halloysite (Gualtieri et al. 2025).

Gualtieri et al. (2024) have recently shown how the 18 FPTI parameters responsible for in vivo adverse effects of mineral fibers are tied to the characteristics of carcinogens KCs used by the IARC to evaluate an agent. Specifically, these KCs are (Smith et al. 2020; Smith 2025):

- KC1: Is it electrophilic, or can it be metabolically activated to electrophiles?
- KC2: Is genotoxic?
- KC3: Alters DNA repair and causes genomic instability?
- KC4: Induces epigenetic alterations?
- KC5: Induces oxidative stress?
- KC6: Induces chronic inflammation?
- KC7: Is immunosuppressive?
- KC8: Modulates receptor-mediated effects?
- KC9: Causes immortalization?
- KC10: Alters cell proliferation, cell death, or nutrient supply?

IARC reviewed all information and data on human carcinogenesis mechanisms and found that *Group 1* agents (like asbestos minerals) show one or more of the 10 KCs (Smith et al. 2016).

MATERIALS AND METHODS

Source of the Sample

The Rio Manubiola is a small creek on the right side of the main Taro River, in the Northern Italian Apennines. In this area, the main outcrops belong to the External Ligurian Units (EL) formed by Mesozoic remnants of the Ligure-Piemontese oceanic basin and its transition to the Adria continental margin (Marroni et al. 2002). These rocks derive from the oceanic crusts (ophiolites; e.g., the Groppo di Gorro ophiolite adjacent to the Rio Manubiola Valley) and their overlying sediments. Typically, the ophiolites of the EL are found as tectonic slices and slide blocks within sedimentary melanges and the sedimentary deposits of the Upper Cretaceous, due to their incorporation in the accretionary prism (Andreozzi et al. 2002; Marroni et al. 2002). An excerpt from the 1:250 000 geological map of the region, depicted in Figure 1, illustrates the distribution of ophiolites in the area, which are grouped under the EL_{or} lithostratigraphic unit, while the Upper Cretaceous sedimentary mélange is reported as EL_{cb} and EL_{pf}. Further details regarding each lithostratigraphic unit can be found in Online Materials¹ Table S1. The ophiolites outcropping along the Rio Manubiola are mainly serpentinites with subordinate basalts. The serpentinites occur both as massive or intensively fractured rocks with dark green to violet appearance, deeply transformed from the original peridotitic protholith. In some outcrops, monogenic serpentinite, basaltic, or polygenic breccias interpose between serpentinites and sedimentary rocks. Portions of ophiolite rocks (especially basalts, basaltic and polygenic breccias) are frequently characterized by an intense hydrothermal activity that creates a pervasive network of secondary veins typical of the so-called opihalcite.

This hydrothermal activity is associated with low-grade (greenschist- to subgreenschist-facies) metamorphism in oceanic environment (Montanini et al. 2008), as well as burial-diagenetic and syn-orogenic conditions (Zaccarini et al. 2008). Typical minerals formed by the interaction between the basalt and hydrothermal fluids include calcite, datolite, prehnite, and quartz. Additionally, clay minerals commonly form through hydration of the basaltic glass (Humphris and Thompson 1978; Bartoli et al. 2008; Zaccarini et al. 2008).

The sample analyzed in this study derives from a localized outcrop of opihalcite identified within the Rio Manubiola streambed. This opihalcite is interpreted to have formed from a serpentinite polygenic breccia and occupies the contact zone with a broader serpentinite exposure in the area (Adorni 1993). The outcrop is partially concealed by overlying slope deposits, which also contain large, meter-sized opihalcite blocks. Secondary vein mineralization within this opihalcite is characterized by major calcite and datolite, with accessory prehnite.

Rare occurrences of fibrous pectolite veins are also present within the selected opihalcite blocks.

Figure 2a displays a polished section of a rock specimen from the outcrop, showing the hydrothermal veins that host datolite fibers. The fibrous morphology of datolite is further evidenced in Figure 2b, while the rock and mineral assemblage of the opihalcite is shown in Figures 2c and 2d and Online Materials¹ Figure S4. Within the veins, which are predominantly composed of bulky datolite, fibrous datolite appears in small vugs. The fibers sometimes exhibit radial growth from the vein walls toward the vein core, which is sealed by coarse-grained datolite and/or calcite (Figs. 2c and 2d).

Another rock specimen deriving from the Rio Manubiola bed at the same locality resembles a thick fibrous datolite vein (5 cm thick, Fig. 3). However, as discussed later, it is mostly composed of pectolite together with datolite and quartz.

Pure specimens of fibrous datolite (FD) and pectolite (FP) used for further characterizations were extracted from the rock samples with the aid of an optical stereoscope.

Sample Characterization

X-ray powder diffraction (XRPD) data for qualitative analyses of the fibrous minerals were collected with an in-house instrument (PANalytical 'X'Pert Pro Diffractometer with a real-time multiple strip detector). Phase identification was performed using the X'Pert High Score Plus (v. 2.2c) software (PANalytical BV, Almelo, The Netherlands). Samples for these analyses were prepared by manually grinding selected fibers in an agate mortar to achieve an average grain size of approximately 10 μm . FD powder was applied as a thin layer to a zero-background sample holder, whereas FP powder was side-loaded into a zero-background sample holder to ensure sufficient material for analysis. Data were collected at ambient conditions using a θ - θ Bragg-Brentano geometry. The specific conditions were: CuK α radiation, 40 kV, 30 mA, range of the data collection = 3–50 $^{\circ}2\theta$, virtual counting time = 100 s/0.0167 $^{\circ}2\theta$ step, 1/2 $^{\circ}$ divergence slit, 1/2 $^{\circ}$ anti-scatter slit, Ni filter on the primary beam, anti-scatter blade slit of 5 mm before the detector, and 0.04 rad Soller slits on both the primary and diffracted beam.

The same FD powder specimens used for XRPD measurement were employed for simultaneous thermogravimetric and differential thermal analyses (TGA-DTA). Data collection was achieved using a Seiko SSC 5200 thermal analyzer operating in air. Samples were heated from 25 to 1000 $^{\circ}\text{C}$ at 10 $^{\circ}\text{C min}^{-1}$. Data acquisition for both TGA and DTA signals occurred every 0.5 s, with α -alumina powder serving as the DTA reference.



FIGURE 1. Geological map of the Rio Manubiola Valley (“Geo-server of the Emilia Romagna Region consulted through the MOKA system” n.d.). The EL_{or} lithostratigraphic unit corresponds to the outcrop investigated in this study. Label descriptions are provided in the supplementary material (Online Materials¹ Table S1), with a full description available from Conti et al. (2020). The star identifies the sampling point. (Color online.)

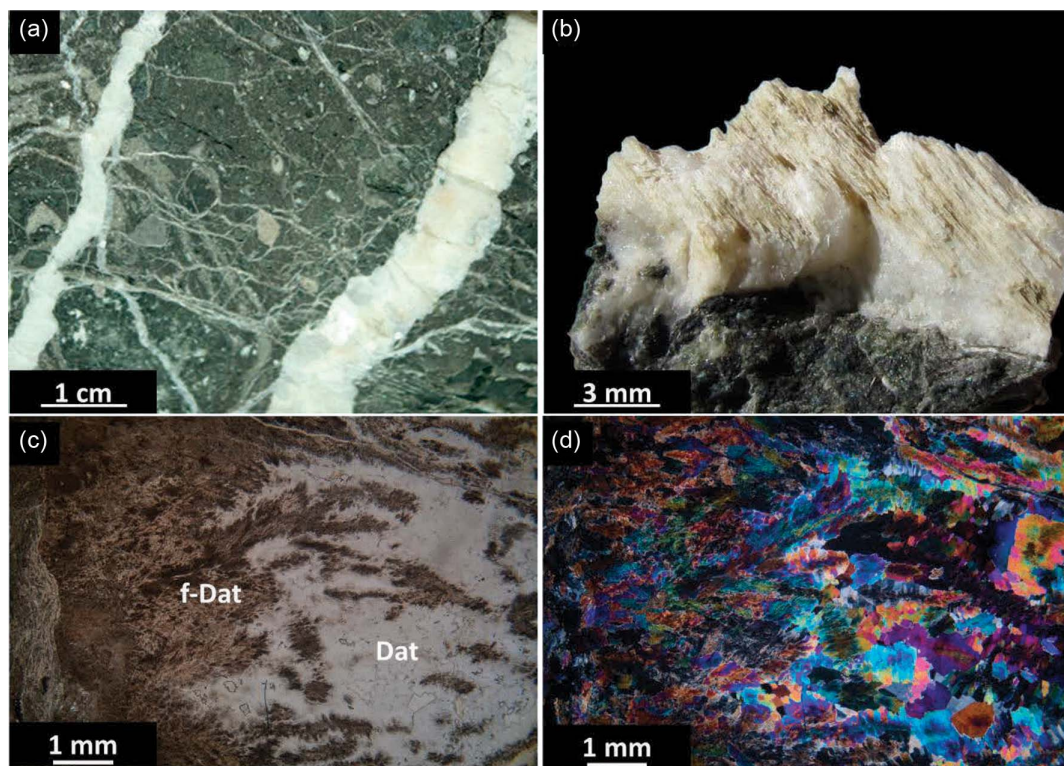


FIGURE 2. Specimens from Rio Manubiola Valley. (a) Polished section well displaying the hydrothermal veins hosting datolite fibers; (b) detail of fibrous datolite; (c) thin section in plane-polarized light showing datolite fibers (f-Dat) growing from left to right in coarse-grained datolite (Dat) vein; (d) same as (c) but in cross-polarized light. (Color online.)



FIGURE 3. Pectolite specimen from the Rio Manubiola bed. (Color online.)

Quantitative determination of major and minor elements in FD was accomplished using electron probe microanalysis (EPMA) at the joint laboratory (LaMa) of the DST and CNR-IGG of Florence (Italy). A rock fragment containing the fibers was embedded in a resin and polished progressively, transitioning to finer abrasives until reaching a 1 μm abrasive. The instrument used was a JEOL Superprobe JXA-8230 equipped with five wavelength-dispersive spectrometers operating with different diffracting crystals and detectors (gas flow and sealed Xe type). Each spectrometer holds two or four diffracting crystals with specific resolution and sensitivity, dedicated to the analysis of specific elements: spectrometer 1 holds TAPJ, LDE1, PETJ, and LDEB; spectrometer 2 holds LIFL and PETL; spectrometer 3

holds LIFH and PETH; spectrometer 4 holds LIF and PETJ; spectrometer 5 holds TAPJ and LDE2. The analytical conditions were 15 kV accelerating voltage, 10 nA beam current, and 3 μm beam diameter. A ZAF data correction model was used to extract quantitative results. Counting times for major elements were 15 s on top of the peak and 7 s for the two tails of the background (apart from Na, 10 and 5 s, respectively); for minor and trace elements, they were 30 s on the peak and 15 s for both backgrounds. F was counted on LDE1 crystal, considering 30 s on peak and 30 s on lower background. Standardizations were performed on Astimex and Smithsonian (Fe and Ti) reference materials. The measured compounds were: SiO_2 , TiO_2 , Al_2O_3 , Cr_2O_3 , FeO , MnO , MgO , CaO , Na_2O , K_2O , BaO , F , B_2O_3 . Chemical data of fibrous datolite from EPMA were rescaled to 100%, considering only elements with counts above the detection limit of the measurement and the H_2O content obtained from TGA measurement.

The crystal structure of FD was determined using synchrotron XRPD data collected at ID22, the high-resolution XRPD beamline (Fitch et al. 2023) of the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. High-resolution XRPD patterns were collected in transmission mode over the 3–30 $^\circ 2\theta$ range [wavelength = 0.427715(5) $\text{\AA}$] using a multi-analyzer stage of 13 silicon (111) crystals coupled to a Dectris Eiger2 CdTe pixel detector (Dejoie et al. 2018). Rietveld refinement of the crystal structure was performed using the GSAS package (Larson and Von Dreele 2004) and its graphical interface EXPGUI (Toby 2001), starting from the crystallographic model of Valmozzola (Parma, Italy) datolite (Rinaldi et al. 2010). A few diffraction peaks not indexed by the starting model indicate the occurrence of contaminants that were revealed thanks to the very high resolution of the XRPD pattern. The non-indexed peaks were marked as “excluded regions” and thus not considered in the refinement. The Rietveld refinement was performed in the $P2_1/c$ space group. The refined parameters were: cell parameters; coefficients of the function modeling the background (a 24-term Chebyshev polynomial); pseudo-Voigt function modeling the profile function of the Bragg peaks (Thompson et al. 1987) with peak intensity cut-off at 10^{-5} of the peak maximum intensity; the overall scale factor. The scattering curves of Si^{4+} , O^{2-} , Ca^{2+} , B, and H were used for the corresponding species; the scattering curve of B was adopted since no X-ray scattering factors are available

for B^{4+} in the GSAS software. Isotropic thermal displacement parameters were refined independently for each atomic site. Fractional coordinates were refined for all atomic positions except H. Indeed, H fractional coordinates and thermal parameter were kept fixed at the values of the starting model (Rinaldi et al. 2010) since these are not refined robustly from powder diffraction data.

Morphometrical and chemical properties of FD were analyzed using a FEG-SEM FEI Nova Nano-SEM 450, equipped with a Bruker XFlash-5010 EDS detector. Samples were prepared by gently crushing the coarse aggregates in an agate mortar, followed by dispersing ~10 mg of the ground material in a 0.1% aqueous solution of AEROSOL OT surfactant. The resulting fiber dispersion was sonicated for 10 min. Four drops of this dispersion were then deposited on a conventional SEM stub and gold-coated after drying. The morphometrical analysis was then performed by observing and measuring about 100 objects (fibers and bundles of fibers) at a magnification of $3000\times$ (field of $138 \times 92 \mu\text{m}^2$) in the secondary electron mode. Object length (L) and diameter (dp) were measured. A regular grid with 0.7 mm shift horizontally and vertically was used for sampling the stub surface to improve statistics and to avoid observing the same objects multiple times.

The Zeta potential of FD and FP specimens was determined in Milli-Q water at various pH values using a Zetasizer Nano Series instrument (Malvern Analytical). Measurements were conducted at 25 °C. Sample dispersions were prepared by finely grinding the fiber specimens in an agate mortar, followed by dispersion in ultrapure water (0.1 wt% solid content). Each dispersion was sonicated in an ultrasonic bath for 15 min and subsequently continuously stirred. The pH was monitored and adjusted to the desired value by adding drops of diluted HCl or NH_4OH (typically 3%). Datolite Zeta potential was measured at pH 4 and 7. For the FP dispersion, the pH was initially adjusted from 9.1 down to 2.3, then incrementally increased up to 10.7, with Zeta potential measurements performed at each step. Prior to data collection, stirring was ceased for 3 min to allow oversized particles to settle. At specific pH values, the Zeta potential was measured five times on the same specimen, and this measurement was replicated on at least two distinct specimens. The results, displayed as the mean of all measurements with their respective standard deviations, will be subsequently presented and discussed in the context of the FPTI model.

Determination of the Toxicity/Carcinogenicity Potential Using the FPTI Model

The FPTI model (Gualtieri 2021) was used to calculate the toxicity/carcinogenicity potential of the fibrous datolite sample. As explained in the introduction section, the model considers morphometric, chemical, and biodegradability-related parameters, and surface reactivity [see Gualtieri (2018) for a detailed description]. A value is assigned to each parameter, depending on its susceptibility to prompt adverse effects. Because the parameters of the model can correlate with each other, a hierarchical scheme that considers cross-correlations is also applied (Gualtieri 2018). The code that defines the hierarchy of the parameter is H , and the weight applied with respect to H is defined as $W_1 = 1/H$, with $H = 1, 2$, or 3 . For example, the surface area has $H = 2$ because it depends on the length ($H = 1$) and diameter ($H = 1$) of the fiber. Another weighting scheme considers the uncertainty in the evaluation of a specific parameter and is defined by the parameter U ($1 = \text{low}$ to null uncertainty, $2 = \text{some degree of uncertainty}$, $3 = \text{high uncertainty}$). The related weight is defined as $W_2 = 1/U$. The FPTI score of each fiber is the sum of all individual FPTI_{*i*} values, classified and weighed according to the equation (Gualtieri 2018):

$$\text{FPTI} = \sum_{i=1}^n W_1 W_2 T_i$$

with $W_1 = 1/H$, the weight of the parameter according to its hierarchy H ; $W_2 = 1/U$, the weight of the parameter according to the uncertainty U of its evaluation; T_i = the class value of the parameter i of the model. The FPTI model is implemented in WebFPTI, a web-based application (Gualtieri et al. 2021). This software is publicly accessible online at <https://fibers-fpti.unimore.it/FPTI/>. A comprehensive manual (WebFPTI_Manual_version4c_250205), available in PDF format, details the methodology for calculating errors associated with each model parameter.

RESULTS AND DISCUSSION

Qualitative Mineralogical Analysis

Qualitative mineralogical investigations were performed on both FD and FP samples. FP was further investigated because

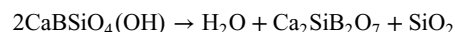
it may be confused with datolite and its characterization is mandatory for the correct separation of the two phases. The XRPD patterns of the two samples in the 5–35 °2 θ interval are shown in Online Materials¹ Figure S5. The FD is mainly constituted by datolite with minor calcite and chlorite. The FP shows dominant pectolite with well-represented quartz and datolite. Several tests were performed to separate fibers of diverse shades of color in the FP sample, trying to obtain pure datolite, but only quartz seems to concentrate in a darker hazelnut-brown specimen, while the intensity ratio of datolite and pectolite remains mostly the same, suggesting an intimate intergrowth of the two phases (Online Materials¹ Fig. S6).

Due to the polyphasic nature of the FP sample, further analyses were performed only on the FD sample.

Thermal Behavior of FD

Thermogravimetric analysis (Online Materials¹ Fig. S7) identifies three distinct reaction ranges: 25–625, 625–800, and 920–1000 °C. The third range is discernible solely from the DTA curve. Within the 25–625 °C interval, a 1.55 wt% mass loss is observed, which can be attributed to the release of surface-bound water molecules (label A in Online Materials¹ Fig. S7) and chlorite hydroxyls. This latter effect is characteristic of chlorites from sedimentary/hydrothermal environments and indicates structural disorder (Caillère and Hénin 1957; Prieto et al. 1991; Bai et al. 1993; Cruz and Real 1995; Földvári 2011). The presence of chlorite, corroborated by XRPD data, is further supported by a shoulder at ~660 °C (label B). This shoulder partially overlaps with the primary dehydroxylation reaction of datolite (label C), which exhibits a maximum at 735 °C (Tarney et al. 1973; Perchiazzi et al. 2004; Földvári 2011; Frost et al. 2013; Krzhizhanovskaya et al. 2018). In the 625–800 °C range, the total mass change is 5.05 wt%. This value is slightly lower than the calculated mass loss for pure datolite (5.63 wt%), likely due to the combined contributions of chlorite and other impurities with differing thermal behaviors.

The dehydroxylation is accompanied by a structural change, where the original structure converts to a tetragonal boron-containing analog of the melilite structure (Tarney et al. 1973; Perchiazzi et al. 2004; Frost et al. 2013). This transformation aligns with the following reaction:



However, Krzhizhanovskaya et al. (2018) proposed a more complex dehydration-induced phase transformation, leading to the formation of melilite (with okayamalite structure, the boron-dominant analog of gehlenite), wollastonite, and minor CaB_2O_4 . The subsequent endothermic events between 920 and 1000 °C, peaking around 965 °C (label D), are attributed to the thermal decomposition and melting of melilite and CaB_2O_4 .

Chemical Composition of FD

The chemical composition of fibrous datolite determined from EPMA is quite close to the ideal formula, and can be averaged as $\text{Ca}_{1.0}\text{B}_{0.999}\text{Si}_{1.021}\text{O}_4(\text{O}_{1.150}\text{H}_{1.199})$. Chemical data of several analytical spots are reported in Table 1. The compounds TiO_2 , Cr_2O_3 , FeO , MnO , and BaO were below the detection limits of the measurements and are therefore not reported in the table.

TABLE 1. Chemical composition of datolite from electron probe microanalysis using quantitative wavelength-dispersive spectrometry

Oxide wt%					
Spot	1	3	3	4	5
SiO ₂	36.84	37.67	37.89	38.18	36.78
B ₂ O ₃	22.37	20.83	21.23	20.48	21.35
CaO	33.93	34.62	34.01	34.46	34.31
Al ₂ O ₃	0.07	0.06	0.08	0.06	0.66
MgO	0.01	0.02	—	—	0.09
Na ₂ O	—	—	—	—	0.06
K ₂ O	—	—	—	0.01	0.01
F	0.17	0.20	0.20	0.20	0.15
H ₂ O	6.6	6.6	6.6	6.6	6.6
SUM	100.00	100.00	100.00	100.00	100.00
Formulas based on one Ca atom					
Si	1.013	1.015	1.040	1.034	1.000
B	1.062	0.969	1.006	0.958	1.002
Ca	1	1	1	1	1
Al	0.002	0.002	0.003	0.002	0.021
Mg	0.001	0.001	—	—	0.004
Na	—	—	—	—	0.003
K	—	—	—	—	0.001
F	0.015	0.017	0.017	0.017	0.013
H	1.211	1.187	1.208	1.192	1.198
O	5.229	5.082	5.196	5.104	5.140

Note: Five analytical spots are reported to show the variability. H₂O content is from TGA.

Crystal Structure of Fibrous Datolite (FD)

Crystal structure refinement (Table 2) of FD does not display peculiar structural features compared to non-fibrous datolite. The observed and final calculated patterns are shown in Figure 4, whereas Tables 3 and 4 report the structural details (atomic coordinates, site occupancy, and atomic thermal displacement

parameters) and bond distances and angles, respectively. Projections along *a*, *b*, *c*, and an oblique projection of the refined crystal structure are shown in Figure 5.

The average refined bond distances of Si and Ca in fibrous datolite is slightly shorter than the datolite from Valmozzola (Rinaldi et al. 2010): 1.6232(12) vs. 1.6327 Å for Si and 2.4677(10) vs. 2.4856 Å for Ca. Instead, the bonding distance for B is longer: 1.5098(16) vs. 1.4808 Å.

By looking at the final fit in Figure 4, it is evident from the difference curve that some misfits are present. In particular, some calculated peaks are highly underestimated compared to their observed intensity and, by looking at their Miller indices, most of them are of the (*0kl*) type [e.g., (011), (020), (031), (014), (032), (034), (025)]. Application of models for the correction of texture effects (e.g., using spherical harmonics) was deliberately discarded because the observed misfit may offer additional insights into fibrous datolite. Indeed, we are measuring a fibrous sample in a capillary setup, and it is likely that some preferred orientation of fibers along the capillary axis occurred. It was shown that in satin spar gypsum (a fibrous variety of gypsum), only reflections of planes parallel to the fiber axis are detected when the fiber axis lies in the horizontal diffusion plane of a Bragg-Brentano geometry (Finkenstadt and Millane 1998;

TABLE 2. Crystallographic data and refinement parameters for datolite

Crystallographic data	
Space group	<i>P</i> 2 ₁ / <i>c</i>
<i>a</i> (Å)	4.83679(1)
<i>b</i> (Å)	7.61346(2)
<i>c</i> (Å)	9.63754(3)
β (°)	90.156
<i>V</i> (Å ³)	354.898(1)
Refinement parameters	
No. of data points	11619
No. of Bragg reflections	698
refined parameters	63
reduced χ ²	2.156
R _{wp} (%) ^a	12.31
R _p (%) ^a	8.58
R (F ²) ^a	9.89

^aAgreement factors for the Rietveld refinement (Larson and Von Dreele 2004).

TABLE 3. Crystal structure coordinates (*x/a*, *y/b*, *z/c*), site occupancy factor (*F*), and atomic displacement parameters (Å²) of fibrous datolite from Rio Manubiola

Label	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	<i>F</i>	U _{iso}
Ca	0.0079(2)	0.6062(1)	0.1631(1)	1	0.0037(3)
B	0.5652(7)	0.0896(5)	−0.1610(4)	1	0.003(2)
Si	0.4690(3)	0.2667(2)	0.0833(2)	1	0.0039(4)
O1	0.2413(5)	0.4013(4)	0.0357(3)	1	0.0037(9)
O2	0.6743(6)	0.2033(4)	−0.0433(3)	1	0.018(1)
O3	0.6732(5)	0.3356(4)	0.2076(3)	1	0.0056(9)
O4	0.3084(5)	0.0921(4)	0.1417(3)	1	0.016(1)
O5	0.2529(5)	0.0892(4)	−0.1671(3)	1	0.0041(9)
H ^a	0.2070	0.0523	−0.0879	1	0.029

^aHydrogen coordinates and thermal parameter were fixed at values from Rinaldi et al. (2010).

TABLE 4. Atomic bond distances (Å) and angles (°) of fibrous datolite from Rio Manubiola

Si-O2	1.572(2)	B-O2	1.520(3)
Si-O2	1.648(3)	B-O3	1.485(3)
Si-O3	1.637(2)	B-O4	1.523(3)
Si-O4	1.640(3)	B-O5	1.512(3)
average	1.623(1)		1.510(2)
O1-Si-O2	113.5(2)	O2-B-O3	107.2(3)
O1-Si-O3	115.2(2)	O2-B-O4	106.7(3)
O1-Si-O4	107.3(2)	O2-B-O5	112.0(3)
O2-Si-O3	105.8(2)	O3-B-O4	108.1(3)
O2-Si-O4	107.7(2)	O3-B-O5	108.7(3)
O3-Si-O4	107.1(2)	O4-B-O5	113.8(3)
average	109.2(7)	average	109.3(1)
Ca-O1	2.286(3)		
Ca-O1	2.262(3)		
Ca-O2	2.410(3)		
Ca-O3	2.656(3)		
Ca-O3	2.640(3)		
Ca-O4	2.430(3)		
Ca-O5	2.640(3)		
Ca-O5	2.507(3)		
average	2.468(1)		

Note: A weighted average was computed, using the inverse square of the standard deviations as weights.

Millane and Dorset 2006; Wang et al. 2024). Thus, it is conceivable that having most of the crystallites aligned along the capillary in Debye-Scherrer geometry can lead to an overestimation of reflections parallel to the fiber elongation axis. In this case, datolite fibers should be elongated in the [100] direction, i.e., *a* axis, also coherently with some crystal habits reported in the literature (red box in Online Materials¹ Fig. S2).

Morphometric Analysis

Figure 6 shows a SEM image of FD, while other fibers and measurement results are shown in Online Materials¹ Figure S8. The morphometric analysis based on SEM micrographs of 100 objects showed an average length (*L*) and diameter (*d_p*) of 15.09 μm (s.d. 13.06) and 3.69 μm (s.d. 2.51), respectively. The average aspect ratio (*L/d_p*) was 5.60 μm (s.d. 4.76) (Online

Materials¹ Table S2). Particles were also divided between bundles of fibers and single fibers. The average *L*, *d_p*, and *L/d_p* for bundles were 16.65 μm (s.d. 14.23), 4.39 μm (s.d. 2.40), 4.43 (s.d. 3.61), respectively (Online Materials¹ Table S2). The corresponding values for single fibers were 9.80 μm (s.d. 5.39), 1.32 μm (s.d. 0.82), 9.53 (s.d. 6.05), respectively (Online Materials¹ Table S2).

The relationship between the length and diameter of each analyzed particle is depicted in Figure 7. As can be observed, most single fibers have a diameter (*d_p*) of <3 μm and a length (*L*) exceeding 5 μm. Conversely, most bundles display a *d_p* and *L* > 3 and 5 μm, respectively. The average size (length and diameter-width) of datolite single fibers meets the fiber size criteria defined by the World Health Organization (1997), i.e., length ≥ 5 μm, width ≤ 3 μm, and aspect ratio (length/width) ≥ 3:1.

Toxicity/Carcinogenicity Potential Using the FPTI Model

Table 5 presents all parameters of the FPTI model, including the hierarchy factor (*H*), uncertainty factor (*U*), classes, and normalized FPTI_i scores. Fibrous datolite yielded an FPTI score of 1.50(0.1), a value that stands as the lowest ever reported for fiber minerals in the existing literature. For comparison, IARC *Group 1* carcinogenic amphibole asbestos fibers have scores between 2.88(0.13) and 3.17(0.13) (Gualtieri 2018), erionite between 2.28(0.21) (Gualtieri et al. 2016) and 2.48(0.05) (Giacobbe et al. 2023), and chrysotile between 2.22(0.07) (Pollastri et al. 2016) and 2.35(0.06) (Di Giuseppe et al. 2021a). In contrast, non-hazardous *Group 3* fibers such as sepiolite and wollastonite have scores of 1.68(0.08) (Gualtieri 2018) and 1.92(0.14) (Di Giuseppe et al. 2021b), respectively. Additional comparative values are available in Table 2 of Gualtieri and Leoncini (2025).

The rather short and thick datolite fibers (*L* = 9.80 μm and *d_p* = 1.32 μm; see Online Materials¹ Table S2; Fig. 7) determine small values of the single FPTI scores for the morphometric parameters (1,1) and (1,2). In fact, shorter fibers can be more

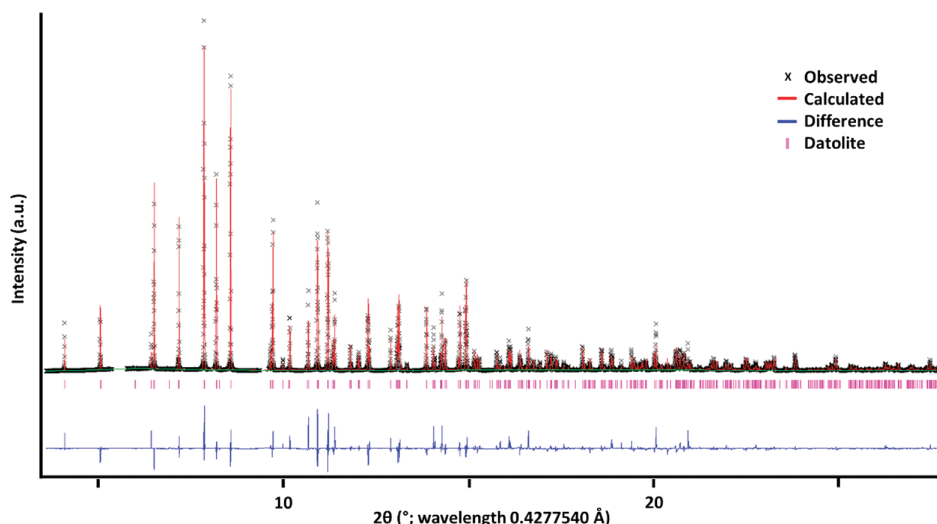


FIGURE 4. Graphical output of Rietveld refinement of XRPD data. Some regions were excluded due to the presence of minor impurities (see relevant experimental section). (Color online.)

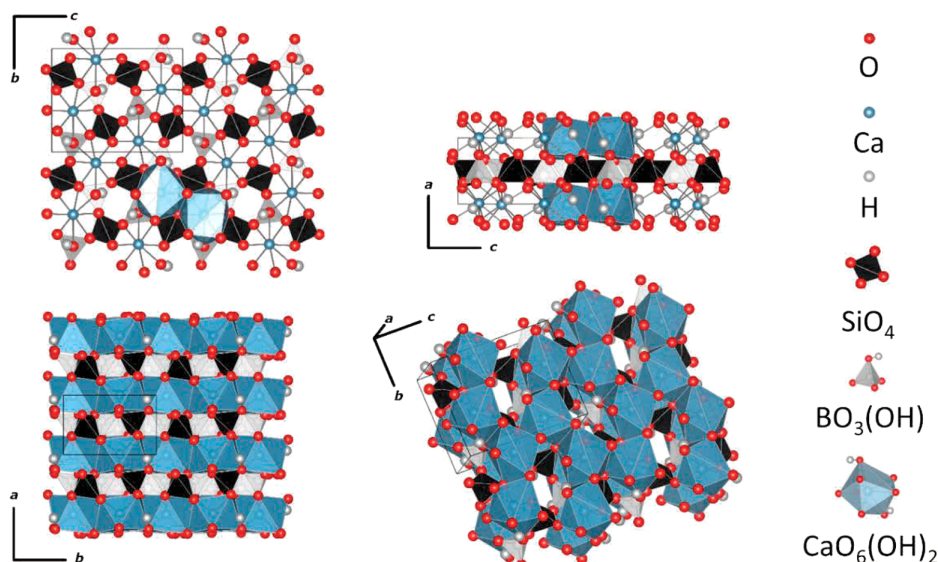


FIGURE 5. Crystal structure of fibrous datolite. In the two upper views, only some Ca-coordination polyhedra are shown. (Color online.)

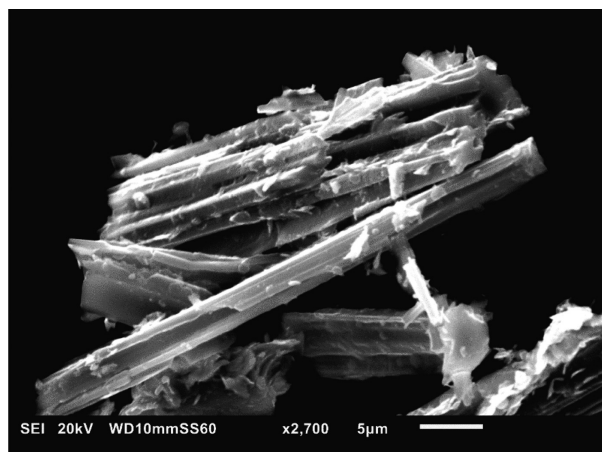


FIGURE 6. SEM image of fibrous datolite.

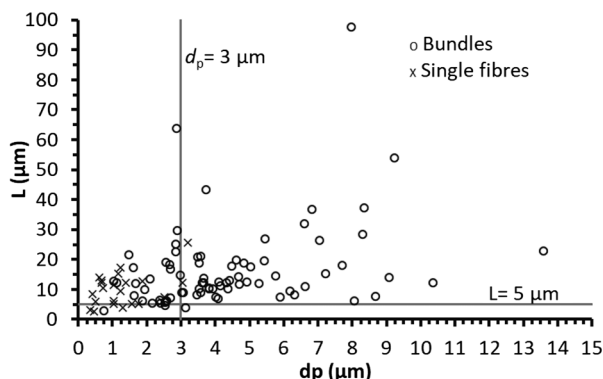


FIGURE 7. Plot of diameter and length of all the fibrous datolite particles employed for morphometrical analyses as measured by SEM. Lines for $d_p = 3 \mu\text{m}$ and $L = 5 \mu\text{m}$ are according to the World Health Organization (1997) fiber size criteria.

effectively removed (clearance) by alveolar macrophages (Stanton et al. 1981; Donaldson et al. 2010), and thicker fibers are less prone to pass through the upper respiratory filters and reach the deep alveolar system, where they eventually prompt adverse effects.

A small value for parameter (1,3) is assigned to flat fiber surfaces, which are known to favor protein adsorption (Lynch and Dawson 2008); this enhanced adsorption can prompt protein-mediated biochemical mechanisms that detrimentally counteract the fiber surface activity.

The acicular/needle-like shape of the datolite fibers [parameter (1,4)] is a detrimental factor of the model because such an aerodynamic shape favors the translocation through the upper airways system and the deposition in the deep respiratory system (Evans et al. 1973). On the other hand, the rather low fiber density [parameter (1,5)] is a positive factor because it reduces the deposition depth of the fibers in the respiratory system (Gualtieri 2018).

The hydrophilic character of the surface [parameter (1,6)] of datolite is another critical parameter because hydrophilic surfaces adsorb biopolymers less effectively than hydrophobic surfaces and are less prone to cell uptake (Gualtieri 2018).

The specific surface area calculated geometrically from the measured mean values of the length $L = 9.80 \mu\text{m}$ and width $d_p = 1.32 \mu\text{m}$ (Online Materials¹ Table S2) and density is $1.1 \text{ m}^2 \text{ g}^{-1}$. This small specific surface area does not accelerate the dissolution of the datolite particles, and the relative parameter (1,7) is set to a maximum value of 0.2. It should be remarked that datolite at $\text{pH} = 4$ is chemically unstable with a fast dissolution rate (see below). For this reason, variations in the surface area actually do not significantly affect the dissolution rate.

Datolite does not contain iron or metals [parameters (1,8), (1,9), (1,10), (1,11) and relative iron/metals release parameters (1,13) and (1,15)] that prompt Fenton primary production of cyto-/geno-toxic hydroxyl radical $\text{HO}\cdot$ species (Donaldson et al.

TABLE 5. The parameters of the FPTI model for fibrous datolite from Berceto (Parma, Italy)

Parameter	H	U	Classes	Normalized score $FPTI_i$	Fibrous datolite
(1, 1) mean fiber length L (μm)	1	1	$L \leq 5.0$	0.0	0.1
			$5.0 < L \leq 10.0$	0.1	
			$10.0 < L \leq 20.0$	0.2	
			$L > 20.0$	0.4	
(1, 2) mean fiber width D (μm)	1	1	$D > 3.0$	0.0	0.1
			$1.0 < D \leq 3.0$	0.1	
			$0.25 < D \leq 1.0$	0.2	
			$D \leq 0.25$	0.4	
(1, 3) crystal curvature	1	2	flat surface	0.05	0.05
			altered surface	0.1	
			cylindrical surface	0.2	
(1, 4) crystal habit	1	1	Curled	0.1	0.4
			mixed Curled/acicular	0.2	
(1, 5) fiber density ρ (g/cm^3)	2	1	acicular	0.4	0.1
			$\rho \leq 2.75$	0.05	
			$2.75 < \rho \leq 3.5$	0.1	
			$\rho > 3.5$	0.2	
(1, 6) hydrophobic character of the surface	2	1	hydrophobic	0.05	0.2
			amphiphilic	0.1	
			hydrophilic	0.2	
(1, 7) specific surface area SSA (m^2/g)	2	1	$SSA > 25.0$	0.05	0.2
			$5.0 < SSA \leq 25.0$	0.1	
			$0 < SSA \leq 5.0$	0.2	
			$Fe_2O_3 + FeO = 0.0$ (no iron)	0.0	
(1, 8) total iron content $Fe_2O_3 + FeO$ (wt%)	1	2	$0 < Fe_2O_3 + FeO \leq 1$	0.05	0.0
			$1 < Fe_2O_3 + FeO \leq 10$	0.1	
			$Fe_2O_3 + FeO > 10$	0.2	
			$FeO = 0.0$ (no ferrous iron)	0.0	
(1, 9) ferrous iron FeO (wt%)	2	1	$0.0 < FeO \leq 0.25$	0.05	0.0
			$0.25 < FeO \leq 1.0$	0.1	
			$FeO > 1.0$	0.2	
			$n = 0$ (no iron atoms)	0.0	
(1, 10) nuclearity or iron atoms n	3	2	$n > 2$	0.02	0.0
			$n = 2$	0.03	
			$n = 1$	0.07	
			$\sum_i \frac{C_i}{L_i} = 0.0$ (no metals)	0.0	
(1, 11) content of metals other than iron ^a (ppm/ppm)	1	1	$0.0 < \sum_i \frac{C_i}{L_i} \leq 1.0$	0.1	0.0
			$1.0 < \sum_i \frac{C_i}{L_i} \leq 5.0$	0.2	
			$\sum_i \frac{C_i}{L_i} > 5.0$	0.4	
			$t \leq 1.0$	0.05	
(1, 12) fiber dissolution time t^b (y)	2	1	$1 < t \leq 40.0$	0.1	0.05
			$t > 40.0$	0.2	
			$v_R = 0.0$ (no iron)	0.0	
			$0.0 < v_R \leq 0.1$	0.03	
(1, 13) velocity of iron release v_R^c (wt%/y)	3	1	$0.1 < v_R \leq 1$	0.07	0.0
			$v_R > 1$	0.13	
			$v_S = 0.0$ (no silica)	0.0	
			$0.0 < v_S \leq 0.5$	0.02	
(1, 14) velocity of silica dissolution v_S^d (wt%/y)	3	2	$0.5 < v_S \leq 1.0$	0.03	0.07
			$v_S > 1.0$	0.07	
			$v_M = 0.0$	0.0	
			$0.0 < v_M \leq 1.0$	0.03	
(1, 15) velocity of release of metals v_M^e (ppm/y)	3	1	$1.0 < v_M \leq 10.0$	0.07	0.0
			$v_M > 10.0$	0.13	
			(-) at $\text{pH} = 4.0\text{--}4.5$	0.1	
			(-) at both $\text{pH} = 4.0\text{--}4.5$ and $7.0\text{--}7.4$	0.2	
(1, 16) zeta potential ζ (mV)	1	2	$\zeta > 20.0$ or $\zeta < -20.0$	0.03	0.03
			$10.0 < \zeta \leq 20.0$ or $-20.0 \leq \zeta < -10.0$	0.07	
			$0.0 \leq \zeta \leq 10.0$ or $-10.0 \leq \zeta \leq 0.0$	0.13	
			cation exchange	0.07	
(1, 17) ζ values ^f inducing fibers' aggregation (mV)	3	1	no cation exchange	0	0.0
(1, 18) cation exchange	1	3		FPTI (error) ^g	1.50 (0.1)

^a $\sum_i \frac{C_i}{L_i}$ = sum of the concentrations of heavy metals (Sb, As, Hg, Cd, Co, Cr, Cu, Pb, Ni, Zn, V, Be) C_i in the fiber (ppm) divided by the limit L_i for that metal according to the existing regulatory system (Tóth et al. 2016) except for Be with limit = 0.5 ppm.

^bThe total dissolution time of the fiber calculated in years (y) following the standardized acellular in vitro dissolution model at $\text{pH} = 4.5$ (Gualtieri et al. 2018).

^cTotal content of elemental iron in the fiber (wt%) possibly made available as active iron at the surface of the fiber, divided by the total dissolution time (y) of the fiber.

^dTotal content of Si of the fiber (wt%) divided by the total dissolution time (y) of the fiber.

^eTotal content (ppm) of heavy metals (Sb, As, Hg, Cd, Co, Cr, Cu, Pb, Ni, Zn, V, Be, and Mn) divided by the total dissolution time (y) of the fiber.

^fAt the $\text{pH} = 7.4$ of the extracellular environment.

^gCalculated as reported in the WebFPTI manual freely available at <https://fibers-fpti.unimore.it/FPTI/>.

2010; Tóth et al. 2016). Hence, all those single FPTI values are null.

The dissolution rate [parameter (1,12)] at pH = 4 is calculated for an ideal 0.25 μm thick fiber, following the method reported in Gualtieri et al. (2018). If a fiber dissolves rapidly in lung fluids, its biopersistence is low, and the fiber is considered less harmful than a biopersistent one. There are no specific data on datolite dissolution rates at pH = 4, but literature data show that boron silicates such as danburite and datolite are chemically unstable in HCl, exhibiting selective boron leaching and progressive structural collapse (Yang 1990). For temperatures around 100 °C, HCl digestion of calcined danburite takes about 1 h (Usmonova et al. 2013), and danburite dissolution at 20–80 °C for 15–60 min has been reported (Ashurov et al. 2009). Here, we have assumed a dissolution time of 30 min for an ideal 0.25 μm thick datolite fiber at pH = 4.

The fast dissolution of datolite may leave a B-rich silica residue that may act in vitro/in vivo to the same extent that chrysotile does when leached in intracellular phagolysosome lung fluids, leaving a product with a residual biochemical toxic action (Gualtieri et al. 2019). For this reason, parameter (1,14), related to silica dissolution, has been assigned a value of 0.07.

Measured zeta potential [parameter (1,16)] of datolite was $-13(1)$ mV at pH = 4 and $-26(1)$ mV at pH = 7. Negative values of zeta potential may correlate with phenomena responsible for adverse effects in vivo, and are comparable to those displayed by mineral fibers at pH = 4–4.5 and pH = 7 (Pollastri et al. 2014). Large (<-20) absolute values of the zeta potential indicate particles that are expected to be well dispersed in fluids. When particles have large absolute values of the zeta potential (<-20 or >20), particle agglomeration [parameter (1,17)] is inhibited, and loose particles are more prone to alveolar macrophages engulfment and clearance (Pollastri et al. 2014 and references therein). For this reason, parameter (1,17) is set to a low value (0.03). Data for fibrous pectolite FP have also been collected (Online Materials¹ Fig. S9 and Table S3), as this mineral fiber may be associated with fibrous datolite.

Cation exchange [parameter (1,18)] is obviously not considered here.

IMPLICATIONS

Here, we present the full crystal-chemical and physical characterization of fibrous datolite, an unclassified, unregulated mineral fiber. The results represent another step toward the final goal of identifying all mineral fibers that may have asbestos-like health effects and thus need to be included in exposure assessment and control.

This fiber is scientifically interesting, but lacks industrial relevance and application. Crucially, its predicted null toxicological potential means it could be used as a negative standard for in vitro cell toxicity studies. This aligns with the use of other fibers, such as wollastonite, which is designated a *Group 3* agent: “not classifiable as to carcinogenicity to humans due to inadequate evidence of carcinogenicity in humans and animals” (Schubauer-Berigan et al. 2026).

The size (length and width) of the datolite single fibers makes them respirable according to the World Health Organization

(1997) fiber size criteria [length ≥ 5 μm , width ≤ 3 μm , and aspect ratio (L/w) $\geq 3:1$]. Nevertheless, due to their lack of biodegradability and absence of iron or other metals in the crystal lattice, these fibers exhibit one of the lowest potential toxicity/carcinogenicity scores among mineral fibers ever evaluated from this perspective.

According to the USGS Mineral Commodity Summary 2025, estimated boron production increased in 2024, as well as the consumption of boron minerals, critical commodities in the EU and the U.K., for the production of glass and ceramics, abrasives, cleaning products, insecticides, insulation, and semiconductors. Knowledge of the potential toxicity of boron-containing minerals such as (fibrous) datolite associated with major borate minerals (such as colemanite, ulexite, danburite, etc.) is an important piece of information for safe management of mining activity and environmental protection.

In short, even if fibrous datolite from the Rio Manubiola Valley is neither easily extractable nor available in sufficient quantities for large-scale applications, this locality may provide small specimens (a few hundred milligrams) for preliminary toxicity studies. Then, since datolite is relatively widespread in boron ores, the occurrence of fibrous datolite in other localities may come to light if interest in this material increases. Also, as shown by Barrer and Freund (1974), it may be possible to synthesize fibrous or asbestiform borosilicates, possibly with a datolite structure, to enable large-scale use of this material.

ACKNOWLEDGMENTS AND FUNDING

Eleonora Braschi and Andrea Orlando (CNR, ISTITUTO DI GEOSCIENZE E GEORISORSE, Florence Unit, Earth Sciences Department of the University of Florence, Florence, Italy) are kindly acknowledged for the EPMA analysis. Emanuele Paganelli is acknowledged for the preparation of thin sections of the datolite-rich rock samples. We would like to express our sincere gratitude to the associate editor, Diego Gatta, for his guidance, to Alessandro Cavallo for his valuable review, and to the other anonymous reviewers for their insightful comments and contributions, which have significantly improved our article. This work was funded by: (1) ECOSISTER: National Recovery and Resilience Plan (NRRP), Mission 04 Component 2 Investment 1.5—NextGenerationEU, Call for tender no. 3277 dated 30/12/2021; Award Number: 0001052 dated 23/06/2022; (2) PRIN2022 “SEEDS—Sediments Eco-recycling Exploitation, Development and Sustainability”—Project Code 2022BCL34N, Finanziato dall’Unione europea—Next Generation EU, Missione 4 Componente 1 CUP-E53D23004330006.

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MANUSCRIPT RECEIVED JULY 28, 2025

MANUSCRIPT ACCEPTED OCTOBER 9, 2025

ACCEPTED MANUSCRIPT ONLINE OCTOBER 16, 2025

MANUSCRIPT HANDLED BY G. DIEGO GATTA

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