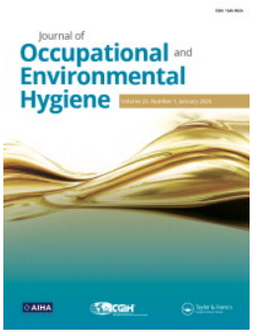

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Enhancing detection of asbestiform minerals in asbestos contaminated talc using FTIR and multivariate data analysis

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ABSTRACT

Talc has been widely used for decades in cosmetic and personal care products because of its unique properties. However, certain talc deposits have historically been found to contain asbestiform minerals, making the detection of these contaminants critical for mitigating asbestos exposure risks in consumer products. To investigate this, laboratory generated mixtures of talc/anthophyllite asbestos and talc/tremolite asbestos were prepared at various concentrations. These mixtures underwent thorough processing with sodium polytungstate (SPT), a heavy liquid, followed by centrifugation, settling, and extraction of separated mineral components. Each extracted sample was analyzed using Fourier Transform Infrared (FTIR) spectroscopy and mineral type was predicted through multivariate data analysis (Partial Least Squares-Discriminant Analysis (PLS-DA)). The PLS-DA model, trained on specific wavenumber regions displaying distinct mineral features, successfully identified anthophyllite and tremolite asbestos in most separated samples. However, in cases where misclassification occurred—where asbestiform minerals were labeled as talc or unassigned—a secondary separation procedure was required. The integration of heavy liquid separation, FTIR spectroscopy, and PLS-DA offers a promising approach for enhancing the detection of asbestiform minerals in asbestos-contaminated talc.

KEYWORDS

Asbestos contamination; Fourier transform infrared (FTIR); heavy liquid separation; partial least squares-discriminant analysis (PLS-DA); talc

Introduction

Talc has been utilized in commercial products, especially in cosmetics and personal care items, due to its unique properties that improve texture and absorb moisture. However, during the mining process, there is a risk of natural asbestos contamination within the talc deposit (IARC 2012). Previous research has reported the presence of asbestiform minerals in consumer products containing talc, raising concerns about chronic exposure through daily use of cosmetics and personal care items (Blount 1991; Gordon et al. 2014; WHO 2014; Fitzgerald et al. 2019; Bloise et al. 2020; Stoiber et al. 2020; Boonruang et al. 2021). The potential contamination of talc with asbestiform minerals poses a significant public health risk, as asbestos exposure has been conclusively linked to severe respiratory diseases, including lung cancer, asbestosis, and mesothelioma with a well-established dose-response relationship (IARC 2012). Therefore, the ability to accurately identify asbestiform minerals in talc-containing products is essential to minimize the risk of asbestos exposure. Various analytical techniques are available for asbestiform mineral characterization, including thermal analysis, transmission electron microscopy (TEM), polarized light microscopy (PLM), Fourier Transform Infrared (FTIR) spectroscopy, as well as X-ray diffraction (XRD) (Bloise 2019). Although FTIR spectroscopy offers advantages such as high sensitivity, rapid measurement, cost efficiency, and nondestructive analysis (Patterson and O'Connor 1966; Gadsden et al. 1970; Beckett et al. 1975; Lee et al. 2024), distinguishing asbestiform minerals from talc remains challenging due to overlapping infrared spectra. However, classification of asbestos may be achievable through the combined use of FTIR and Partial Least Squares-Discriminant Analysis (PLS-DA) (Lee et al. 2024; Alquwayi et al. 2025). This study explicates how to enhance detection of asbestiform minerals in asbestos contaminated talc using FTIR, heavy liquid separation and PLS-DA.

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Methods

Sample preparation

Pure talc (Spectrum Chemical Mfg. Corp.), anthophyllite asbestos (NIOSH), and tremolite asbestos (NIOSH) were used. All materials were sieved to a consistent particle size (<53 µm) using ASTM E-11 specification sieves (No. 270). A mixture of 1 mg of each mineral and 99 mg of potassium bromide (KBr) was prepared for the PLS-DA model training. Laboratory generated talc/anthophyllite and talc/tremolite mixtures were prepared in four concentrations, 2, 1, 0.5, 0.1 wt% of asbestos to talc with total masses of 2, 2, 8, and 20 g, respectively.

Heavy liquid separation

Heavy liquid separation was conducted with sodium polytungstate (SPT, density of 2.89 g/cm^3, GEO liquids) of which density is intermediate between talc (2.83 g/cm^3) and amphibole minerals (2.98 g/cm^3) (Chornkrathok et al. 2024). Before each sample preparation, the SPT density was verified with a portable density meter (Mettler-Toledo, Columbus, OH, USA). Depending on the amount of laboratory generated mixtures of talc and asbestos, 36 mL (for 2.0 and 1.0 wt% mixtures), 72 mL (for 0.5 wt% mixtures), or 144 mL (for 0.1 wt% mixtures) of SPT and mixtures were placed in a laboratory beaker and mixed gently and thoroughly. The mixed sample was divided into centrifuge tubes and centrifugation was performed using a centrifuge (LW Scientific, Lawrenceville GA, US) at 1,534 relative centrifugal force (3,500 rpm) for 60 min to achieve a separation. The heavy materials were allowed to settle inside the centrifuge tubes at least 8 hr. The upper section of each centrifuge tube was removed using a syringe and the bottom section of each centrifuge tube was carefully extracted with an electronic micropipette (50 µL, Eppendorf, Germany). The extracted samples were filtered using 25-mm polycarbonate filters (0.4 µm pore size) and rinsed with distilled water to clean the remaining SPT. The samples were dried in the desiccator for 20 min. The visible samples were scraped off and mixed with KBr for FTIR analysis. The amount of separated material obtained after heavy liquid separation varied depending on the asbestos concentration of each sample. In addition, spectral normalization during PLS-DA data processing was applied to minimize variability related to sample.

FTIR spectroscopy

A FTIR spectrometer (Nicolet iS5, Thermo Fisher Scientific Inc.) equipped with a diffuse reflectance accessory (COLLECTOR II, Thermo Fisher Scientific Inc.) for Diffuse Reflectance Infrared Fourier transform (DRIFT) technique was utilized to obtain 100 spectra for PLS-DA model training and 5 spectra of each heavy liquid separation sample with 4,000 to 400 cm^{-1} wavenumber range at 4 cm^{-1} resolution by averaging 16 scans. An average of 10 blank KBr measurements was used as a background on each measurement day.

Data processing and multivariate data analysis

PLS-DA (PLS_Toolbox, v8.9.1, Eigenvector Research Inc.) operating in MATLAB (The MathWorks, Inc.) was employed to identify asbestiform minerals from the samples. PLS-DA model was developed using specific FTIR spectral regions of 3,640–3,700 cm^{-1} and 700–779 cm^{-1} with 100 FTIR spectra of each pure talc, tremolite, and anthophyllite. This training data, with known classifications, formed the basis for a predictive model to differentiate mineral types in unknown samples. The PLS-DA model utilized two latent variables (LVs) derived from FTIR spectra within specified wavenumber regions, creating a two-dimensional model that accounted for 79.24% of total data variance (43.71% by LV1 and 35.53% by LV2). Data preprocessing included the Savitzky–Golay 1st derivative (second-order polynomial with a 15-wavenumber smoothing window), followed by 1-normalization and median centering for each prediction. Model outputs classified samples into one of the three mineral categories based on the highest assignment probability.

Results

Calibration samples for PLS-DA

FTIR spectra of pure talc, anthophyllite, and tremolite measured in the wavenumber range 400–4,000 cm^{-1} are shown in Figure 1. The spectra show that tremolite and anthophyllite have distinct features from talc in the OH stretching (3,640–3,700 cm^{-1}) (Della Ventura et al. 2018) and the symmetric Si-O-Si stretching (700–779 cm^{-1}) (Campopiano et al. 2015) regions. Anthophyllite has a broad peak around 3,665 cm^{-1} and tremolite has three broad peaks at 3,674 cm^{-1}, 3,658 cm^{-1}, and 3,650 cm^{-1} while talc shows a peak at 3,675 cm^{-1} in the OH stretching region. In the symmetric Si-O-Si stretching region,

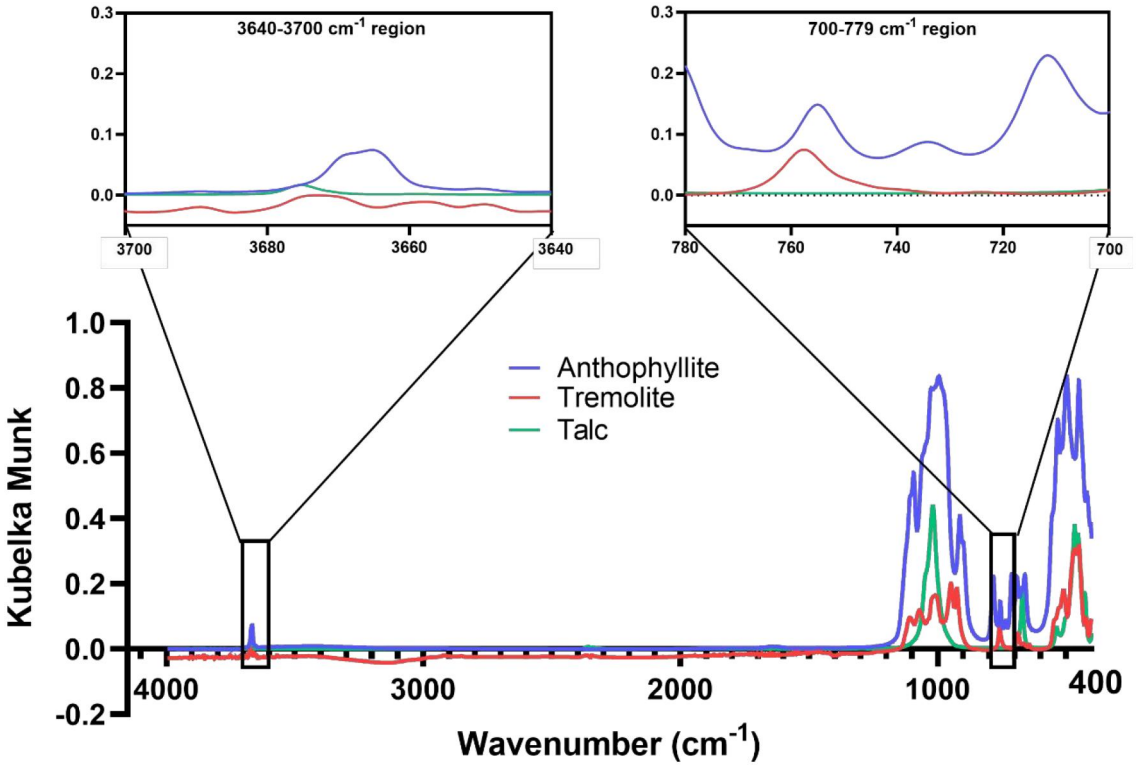

Figure 1. FTIR spectra of pure talc, anthophyllite, and tremolite. Insets indicate the regions of interest to train a PLS-DA model.

talc does not have any feature, however, anthophyllite shows three peaks at 755 cm^{-1}, 734 cm^{-1}, and 711 cm^{-1}, and tremolite has a peak at 757 cm^{-1}. The classification of three minerals with calibration samples in a score plot (left) along with a cross-validation prediction plot (right) with threshold values (dotted line) are shown in Figure 2. Thresholds of anthophyllite, talc, and tremolite are 0.34, 0.58, and 0.25, respectively. The prediction probability of an unknown sample should be above the threshold value to be assigned to a mineral type. The clear distinction between three minerals is shown in the cross-validated prediction plot indicating that asbestos can be identified using the PLS-DA model.

Talc and asbestos mixture samples

Laboratory generated pure talc, mixtures of talc/anthophyllite and talc/tremolite with different concentrations of asbestos (2.0, 1.0, 0.5, and 0.1 wt%) without heavy liquid separation procedure were measured with FTIR as shown in Figure 3. Both talc/anthophyllite and talc/tremolite mixtures showed the dominant peaks located around 3,675 cm^{-1} in OH stretching region associated to talc (Elnazer et al. 2023). PLS-DA classified all lab generated mixtures samples without heavy liquid separation to as talc (Table 1).

It should be noted that the heavy liquid separation was performed with care, as the bottom section of the sample (dense minerals) can be easily contaminated during the extraction process. After heavy liquid separation, IR spectra of separated samples are shown in Figure 4A and 4C. The spectra clearly show that 2.0 and 1.0 wt% have distinct features from talc in both OH stretching and symmetric Si-O-Si stretching regions after separation. For anthophyllite, a broad peak around 3,665 cm^{-1} and small peaks around 755 cm^{-1}, 734 cm^{-1}, and 710 cm^{-1} appeared, while for tremolite, three peaks around 3,674 cm^{-1}, 3,658 cm^{-1}, and 3,650 cm^{-1} and a small peak around 755 cm^{-1} emerged as indicating the features from FTIR spectra of pure materials. The PLS-DA model correctly identified anthophyllite asbestiform mineral with 72% accuracy and tremolite asbestiform mineral with 88% accuracy (Table 1). The second separation was necessary when the model predicted the first separated samples either talc or unassigned (none of talc, anthophyllite, and tremolite found) by the PLS-DA model prediction because it is known that the mixture samples contain asbestos. The second separation improved the identification accuracy of the PLS-DA model to 95% for anthophyllite and 98% for tremolite.

SEM images before (top) and after (bottom) separation for talc/anthophyllite and talc/tremolite mixtures

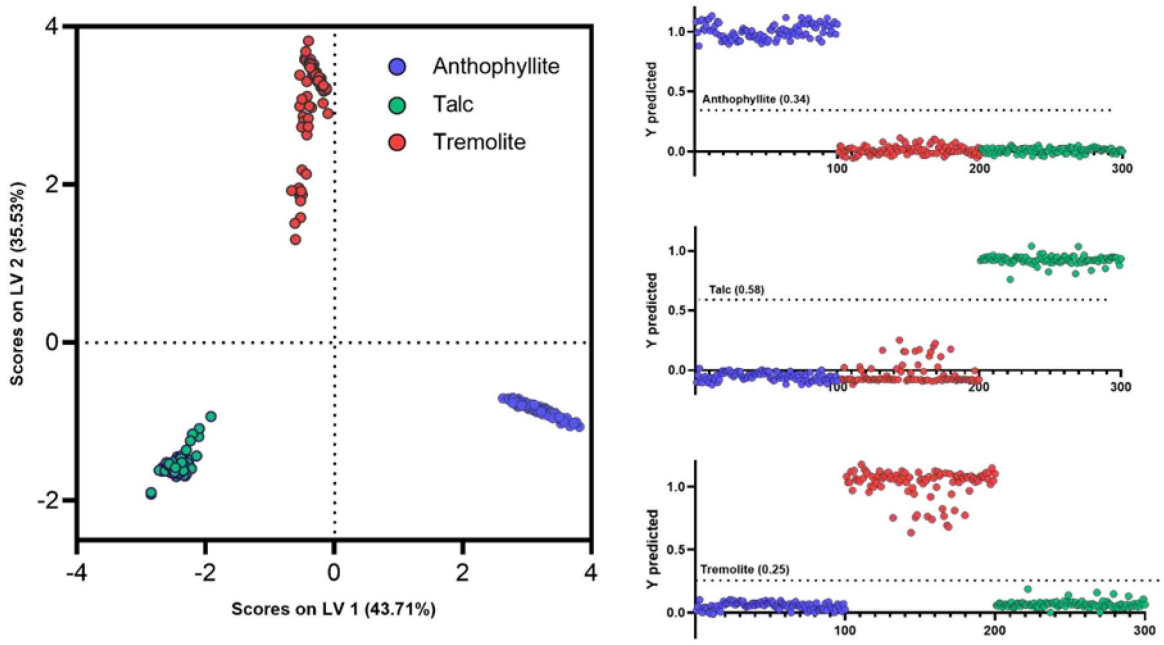

Figure 2. Classification of talc, anthophyllite, and tremolite in the PLS-DA score plot (left) and cross-validated prediction plot with Y-predicted thresholds of each of them (right).

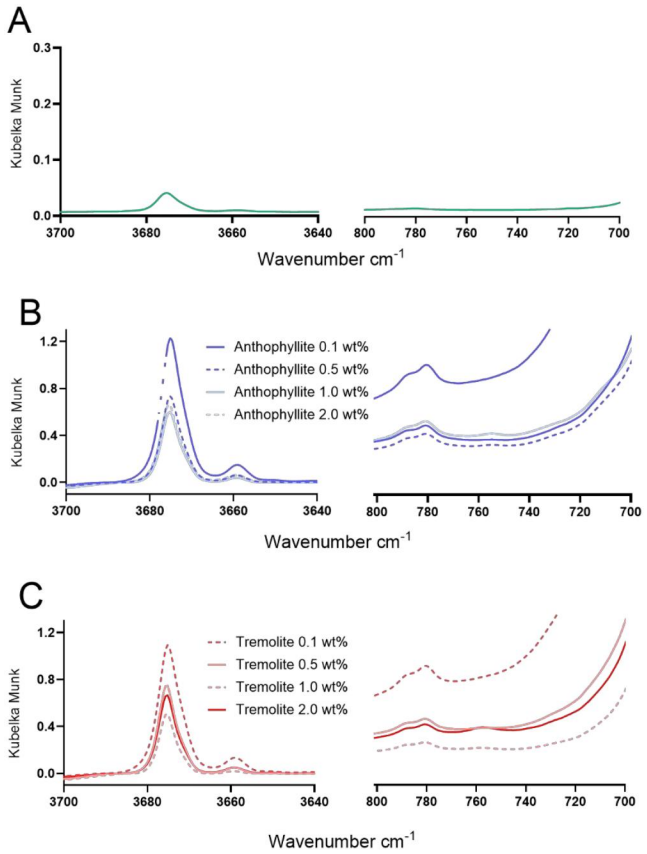

Figure 3. A) FTIR spectra of pure talc, FTIR spectra for 2.0, 1.0, 0.5, and 0.1 wt% of B) anthophyllite and C) tremolite mixed with talc.

are shown in Figure 4B and 4D. In general, large amount of talc particles were removed through the separation process but not completely.

Discussion

This study demonstrated an integrated method combining heavy liquid separation, FTIR spectroscopy, and multivariate analysis for enhanced detection of

Table 1. PLS-DA classification of laboratory generated mixtures of talc/anthophyllite and talc/tremolite without heavy liquid separation (HLS) and after 1st and 2nd heavy liquid separation (HLS).

	Anthophyllite					Tremolite			
	Without HLS	After 1st HLS	2nd HLS	After 2nd HLS		Without HLS	After 1st HLS	2nd HLS	After 2nd HLS
2.0%					2.0%				
#1	Talc	Anthophyllite	N		#1	Talc	Tremolite	N	
#2	–	Anthophyllite	N		#2	–	Tremolite	N	
#3	–	Unassigned	Y	Anthophyllite	#3	–	Tremolite	N	
1.0%					1.0%				
#1	Talc	Anthophyllite	N		#1	Talc	Tremolite	N	
#2	–	Talc	Y	Anthophyllite	#2	–	multiassigned	Y	Tremolite
#3	–	Anthophyllite	N		#3	–	Tremolite	N	
0.5%					0.5%				
#1	Talc	Anthophyllite	N		#1	Talc	Tremolite	N	
#2	–	Anthophyllite	N		#2	–	Tremolite	N	
#3	–	Anthophyllite	N		#3	–	Tremolite	N	
0.1%					0.1%				
#1	Talc	Anthophyllite	N		#1	Talc	Tremolite (3/5)	Y	Tremolite (4/5)
#2	–	Anthophyllite (2/5)	Y	Anthophyllite	#2	–	Tremolite	N	
#3	–	Talc (4/5)	Y	Anthophyllite (2/5)	#3	–	Tremolite	N	

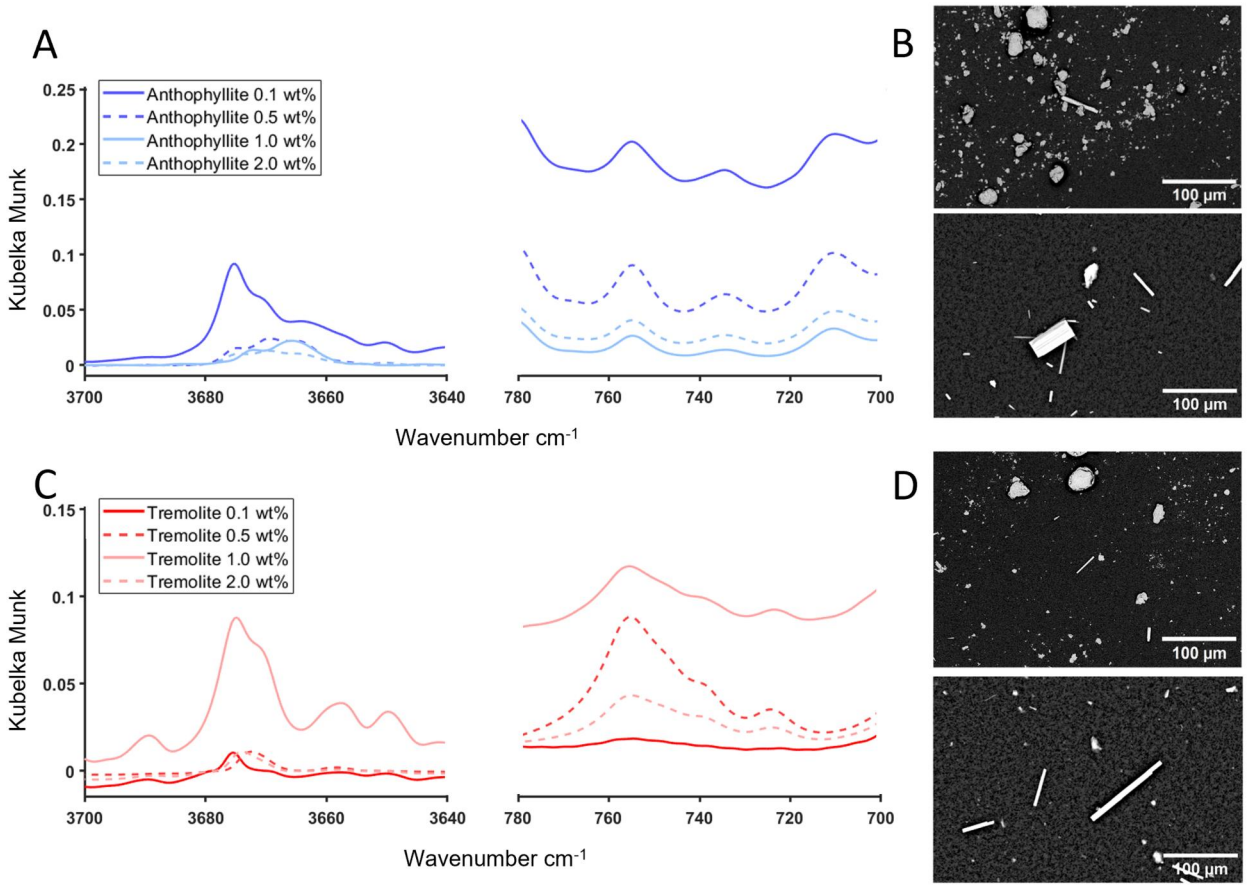

Figure 4. FTIR spectra of 2.0, 1.0, 0.5, and 0.1 wt% of A) anthophyllite and C) tremolite after heavy liquid separation, SEM images of 1.0 wt% B) anthophyllite and D) tremolite before (top) and after (bottom) heavy liquid separation.

asbestos contaminants in talc. Without the heavy liquid separation, the dominance of talc peaks in mixture spectra interfered asbestiform mineral signals and led to misclassification. Heavy liquid separation successfully refined asbestiform mineral content in the separated fraction. It improved FTIR features of asbestiform minerals and PLS-DA model classification. In addition, training of PLS-DA model with specific wavenumber regions of characteristic mineral features was effective in distinguishing asbestiform minerals

from talc under controlled laboratory conditions. Although a direct comparison between the PLS-DA prediction and other asbestos standard analysis methods (PLM or TEM) was not made in the present study, the authors' previous research (Alquwayi et al. 2025) demonstrated comparable results between PLS-DA and PLM analysis of asbestos containing materials in bulk. The combination of heavy liquid separation, FTIR spectroscopy, and multivariate analysis provides a practical way for enhancing asbestiform mineral detection in talc containing products. Improved identification of trace asbestiform minerals in talc could reduce the release of hazardous fibers into the environment, promote safer manufacturing practices, and inform regulatory actions, thereby mitigating exposure risks to consumers and workers.

Limitations

Despite the promising result and applicability of the procedure for identifying asbestiform minerals in contaminated talc, there are some limitations. First, the present study was conducted under controlled laboratory conditions with a small sample number ($n = 24$). A study with large sample number and lower asbestos concentration might be needed to confirm its validation. In addition, the procedure should be validated with the naturally occurred asbestos contaminated talc samples. Second, the bottom section of the sample can be easily contaminated during the extraction procedure, although the upper section of the separated samples was removed with care. Third, it is still unclear how many repetitions of the separation procedure would be necessary when analyzing real samples that contain asbestos at concentrations below the limit of detection.

Conclusion

PLS-DA has been explored as a statistical tool capable of predicting mineralogical compositions to improve the detection accuracy of asbestiform minerals. This study enhances the identification of asbestiform minerals in talc, providing a more robust and reliable approach for public health safety. The laboratory procedure can be an option for identification of asbestos contamination in talc-based consumer products enabling improved protection for consumers from exposure to contaminated talc. Further studies might focus on enhancing sensitivity for trace asbestos detection in a lower asbestos contaminated talc through increasing FTIR spectral resolution, refining spectral feature selection algorithms that can better discriminate overlapping FTIR features, and expanding PLS-DA training model with low-concentration asbestos samples.

Disclosure statement

No potential conflict of interest was reported by the author(s).

Data availability statement

Data will be provided upon reasonable request.

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