BMPC 2017

XVII. Balkan Mineral Processing Congress

XVII BALKAN MINERAL PROCESSING CONGRESS
1 - 3 NOVEMBER 2017 · ANTALYA TURKEY

Proceedings

November 1-3, 2017

Antalya,Turkey

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İSTANBUL TEKNİK ÜNİVERSİTESİ 1773

Turkish Mining Development Foundation

Proceedings of the

XVII. BALKAN MINERAL PROCESSING CONGRESS

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ISBN: **978-975-7946-42-7**

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FOREWORD

Mineral processing is an art of treating raw ores in order to separate valuable minerals from the waste rock as well as preparing mineral products for specific needs. It is the first process that most ores undergo after mining in order to provide concentrated material for the downstream metallurgical processes. The primary operations are comminution and concentration, but there are other important operations in a modern mineral processing plant, including sampling and analysis and dewatering. In mineral processing, a number of unit operations are required to prepare and classify ores before the valuable constituents can be separated or concentrated and then forwarded on for use or further treatment. Mineral processing education at the Istanbul Technical University has started as a division of the "Mining Engineering Department" which was founded as a part of the Mining Faculty in 1953. The division was re-organized as a Mineral Processing Engineering Department of the same faculty in 2007, in order to meet the requirements of engineering education. It is the first and only one in Turkey.

The first Balkan Mineral Processing Congress was held in Varna, Bulgaria in 1973. The “XVIIth Balkan Mineral Processing Congress – BMPC 2017” is organized by ITU Mineral Processing Engineering Department and the Turkish Mining Development Foundation between November 1 and 3, 2017, in Antalya-Turkey. “BMPC 2017” will bring together a large number of academicians from Turkey and many other countries as well as the authorities from the companies that are operating in the mining, mineral processing, and extractive metallurgy industries in order to create an opportunity to evaluate the future of the sector. In this context, BMPC represents a technical platform for the safe and sustainable future of the mining and mineral processing industries in Balkan Countries. We are inviting you to become a part of this global challenge by attending our congress through participation, presentation, exhibition and supporting our activities. In this respect, Antalya with its dynamism and diversity presents a unique opportunity for the attendees. We assure you that we will provide all means to make you enjoy and benefit from this extraordinary event.

The Congress will gather over one hundred professionals and academics from all over the world to exchange knowledge and experience, to present the results of scientific research and to discuss innovations in the mineral processing industry. Universities specializing in mineral processing, a number of important mining companies, major leading providers of services and technologies will participate in the Congress.

We are very pleased to welcome you to attend and we are sure you will enjoy the Congress from both scientific and social points of view. Looking forward to meeting you in Antalya!

Prof. Dr. Fatma Arslan | Prof. Dr. Ayhan Ali Sirkeci

FOUNDATION'S FOREWORD

As Turkish Mining Development Foundation, we are happy to be organizer of 17. Balkan Mineral Processing Congress. I would like to express my sincere thanks to the Balkan Scientific Committee, Turkish Organization Committee, to the Sponsors, exhibitors and all the attendees. Our special gradidute to Istanbul Technical University, Mining Faculty and Mineral Processing Department.

We wish successful and fruitful congress and sunny days in Antalya.

With kind regards,

Prof. Dr. Güven Önal

President of Turkish Mining Development Foundation

Balkan Scientific Committee Member

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PYROPHYLLITE-MINERAL OF THE FUTURE

FOR APPLICATION IN AGRICULTURE

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ABSTRACT

Frequently repeated phrases are that mining is a low-accumulative activity, with high production expenses and low prices of final products, which puts mining at a disadvantage compared to other, less demanding activities. We often point out that employees in the field of mining have been taking into account production expenses much more than generated profit for a long time. At the same time, capacities have not been sufficiently utilised.

Non-metallic mineral raw materials, such as pyrophyllite ($Al_2Si_4O_{10}(OH)_2$), have an important role in economic development, whether as final products or as raw materials in manufacturing in processing and other industries. By its available quantities, pyrophyllite is one of the most important domestic natural non-metallic mineral raw materials in BiH (18.000.000t), [1-2]. There are almost no economic sectors that do not use pyrophyllite. In other words, it is used in the majority of industries, including civil engineering, agriculture, and lately increasingly in technologies for environmental protection. The existence of potential raw material base, pyrophyllite, in significant quantities, provides extensive possibilities for the development of manufacture with a wide product range.

This paper will attempt to point out the possibilities for prospective processing and application of high-quality concentrates of pyrophyllite for new materials with added value, with special emphasis on agriculture.

Key words: geology, deposits, mining, pyrophyllite, agriculture, materials.

1 INTRODUCTION

In recent years, the quantity and quality of materials obtained on the basis of non-metallic mineral raw materials in the most developed countries is three to five times higher than the value of products based on metallic minerals. Therefore, the modern, highly technological civilisation is extremely dependent on a stable and suitable supply of non-metallic mineral raw materials, since they are necessary for the manufacture of materials serving as material components in the manufacture in the majority of industrial activities and in agriculture.

For different domains of processing application in a synthesis of various materials (of defined structure and properties), the same non-metallic minerals may have different properties. Since properties of non-metallic mineral raw materials depend on a crystal-chemical structure, and the structure may be modified by technical processing – natural raw materials and non-metallic mineral raw materials must be technologically treated very seriously, [3].

Namely, for already well known sophisticated use of limited domestic resources of high-quality non-metallic mineral raw materials – a realistic estimate of their availability, it must be taken into account that diverse application of certain non-metallic mineral raw materials depends not only on their basic chemical component, but also on their particular mineralogical and crystallographic properties, which determine their structure and their processing features for the production of various materials.

Therefore, resource and exploitation availability of domestic non-metallic mineral raw materials must, first and foremost, be estimated and identified by the possibility of their application. In developed market economies, risk factors in non-metallic mineral raw materials manufacture and processing industry are analysed exclusively in the domain of marketing. In this sense, marketing does not include activities often implied in our practice: advertising, promotion, distribution of samples, etc. Modern marketing in this domain is a technological, technical and commercial adjustment of non-metallic mineral raw materials manufacturing to market demands (processing domain). In case of this particular mineral, the issue is not with the identification (type, reserves, method of obtainment, rough preparation), since with this level of information, with minimum processing, the mineral may be sold, but at a low price and with marginal profit. Modern and highly profitable use of available (and predominantly non-renewable) mineral resources (for example, in chemical and ceramics industry, pharmacy and cosmetics, metallurgy and civil engineering, development of various materials in agriculture) requires application of higher levels of technological preparation and processing.
Natural non-metallic mineral raw materials can never be used directly in a controlled (modern – sophisticated) interaction: **material synthesis for functions of intended use ↔ defined properties of input raw material component.** For this reason, the modern scientific procedure for fundamental and developmental research sets rational bases for the development of technological operations and processes for preparation and processing of natural non-metallic mineral raw materials to semi-products – products with required properties. Properties of these non-metallic mineral raw material products define and ensure their use as a raw material component in a synthesis of **materials for functions of intended use.**

2 APPLICATION OF PYROPHYLLITE

In the territory of BiH, there are a number of geologically analysed deposits of natural non-metallic raw materials, which may be used in agricultural production for soil improvement (melioration) and enrichment with biogenic elements (fertilisation) both in the conventional (existing) agriculture and in organic agriculture in rural areas. In this sense, natural non-metallic mineral raw materials include: limestone, dolomite, zeolite, bentonite, diatomaceous earth, pyrophyllite, rocks with increased content of useful microelements.

In recent past, the use of some of natural non-metallic mineral raw materials began in improvement of properties of degraded agricultural soils in rural areas, primarily including: limestone, zeolite and peat. However, the latest research of pyrophyllite deposits with their natural properties indicated they could be used in agricultural production (feed, fertilisers, plant protection, fruit growing, viniculture, vegetable growing, floriculture, herbs, forestry, greenhouse production) and in rural areas to:

improve some features of degraded surfaces of agricultural land (meliorative use) and

restore biogenic and necessary mineral substances required for plants (fertilisation use) for a successful development, growth and maximum satisfactory quality yield of cultivated plants.

The use of natural non-metallic mineral raw material (pyrophyllite) in agricultural production will ensure a series of positive effects:

development of conditions for initiation of agricultural plant production (which is also the basis for cattle production) under the principles of organic agriculture, which results in agricultural products of high biological (eco) value, in demand both on our market and on the markets of developed countries;

there is a realistic possibility for a positive foreign trade balance, which creates conditions for increased financial investments in the agricultural production;

the country is relieved of import of certain raw materials (phosphates) for the production of mineral fertilisers, and import of energy sources (natural gas) for the production of certain mineral fertilisers;

employment is increased in the exploitation of pyrophyllite, intended for agricultural production;

rational use of surfaces of degraded agricultural land in agricultural production will be increased;

in addition to the production of biologically more valuable (eco) food in compliance with the principles of organic agriculture, complete environmental protection will also be ensured.

These advantages of pyrophyllite as a natural non-metallic mineral raw material are sufficient reasons for the need to verify their value for the use in agricultural plant production, particularly in compliance with the principles of organic agriculture, and in other industries, including: ceramics and chemical, civil engineering, paint and varnish industry, production of hydro-insulating materials, paper industry, pharmacy and cosmetics, production of plastics and rubber, production of refractory materials, food industry, protection of water and waste water system, decontamination and detoxification of contaminated surfaces, stabilisation of acid soil, etc., [6-8].

3 EXPERIMENTAL RESEARCH RESULTS AND DISCUSSION

All experimental research of dry micronisation of pyrophyllite ($Al_2Si_4O_{10}(OH)_2$) was carried out on a pit sample, applying the latest methods for identification of all required physical, chemical and mineral properties of micronised pyrophyllite products, and for relevant process parameter monitoring.

The deposit of pyrophyllite schist in Parsovići, Konjic, is located around 25 km northwest of Konjic, with 14 km asphalt connection to the highway Sarajevo — Mostar. The deposit in a mountainous area, which is the southwest branch, and the pyrophyllite deposit area altitude ranges between 350 and 600 metres, as presented in Figure 1. Confirmed ore reserves of pyrophyllite schist "Parsovići-Konjic" are 18.000.000 t.

Figure 1. Pyrophyllıte Deposıt, Parsovıćı, Konjıc

The starting sample was homogenised. Using the chess board method, samples were extracted for identification of hygroscopic moisture, chemical ore composition, DTA-TG analysis, FTIR-analysis, cation exchange capacity (CEC), XRD-analysis. In addition, qualitative mineralogical analysis was performed using polarising microscope in released light of the ore sample by classes of size and petro preparation, semi-quantitative chemical analysis of the starting sample of ore-ore preparation on SEM.

3.4.Physical, chemical and mineral sample properties

Granulometric composition of pyrophyllite ore was identified on two samples of pit pyrophyllite ore. The first ore sample was a pit sample crashed to the size class of -5.0+0.00 mm, and the second was obtained by crushing on a roller crusher to the size class of -2.00 mm. During the granulometric analysis of both pyrophyllite samples, all sift-outs at screening, along with the sift-out of the last screening, were measured, information were processed and classified in Table 1.

Table 1. Granulometric Composıtıon Of Pyrophyllıte Ore Sample, -5.00+0.00mm And 2.00 Mm

Size class, mm	M, %	M, %↓	M, %↑	Size class, mm	M, %	M, %↓	M, %↑
- 5.00 + 3.36	0.65	0.65	100.00	- 2.38 + 1.60	0.89	0.89	100.00
- 3.36 + 2.38	0.94	1.59	99.35	- 1.60 + 1.00	1.01	1.90	99.11
- 2.38 + 1.60	1.40	2.99	98.41	- 1.00 + 0.63	1.77	3.67	98.10
- 1.60 + 1.00	2.62	5.61	97.01	- 0.63 + 0.40	3.05	6.72	96.33
- 1.00 + 0.63	3.16	8.77	94.39	- 0.40 + 0.30	3.79	10.51	93.28
- 0.63 + 0.40	5.14	13.91	92.23	- 0.30 + 0.20	4.78	15.29	89.49
- 0.40 + 0.30	5.16	19.07	86.09	- 0.20 + 0.10	19.20	34.49	84.71
- 0.30 + 0.20	4.94	24.01	80.93	- 0.10 + 0.050	10.91	45.40	65.51
- 0.20 + 0.10	11.35	35.36	75.99	- 0.050 + 0.037	0.56	45.96	54.60
-0.10 + 0.00	64.64	100.00	64.64	- 0.037+0.00	54.04	100.00	54.04
Input	100.00	-	-	Input	100.00		

Based on the granulometric composition of both pyrophyllite ore samples, it may be concluded that, regardless of its mineral composition, the ore contains strikingly different mineral raw materials in terms of hardness. In the first case, with the size class -5+0.00 mm, obtained by crushing on a roller crusher, the majority of raw material (64.64 %) was smaller than -0.1 mm. Since the crushing was performed on the roller crusher, with the roller distance of 5mm, with not a large quantity of grit as some other types of crushers (cone and circular), it may be concluded that exceptionally soft minerals include such a large share of crushed class in the raw material, but owing to the presence of larger fractions, there are minerals of great hardness. A similar conclusion refers to the class of -2+0.00mm, which was also obtained from the roller crusher. In this case as well, the granule composition shows a very large share of a very fine class of -0.037+0,00mm (54.04%), and almost the same share of the -0.1+0.00 in this sample (65.51%), as with the larger sample of 64.64%.

Chemical properties, i.e. identification of chemical composition of the starting pyrophyllite sample was made using the standard analytical method, and the analysis results are presented in Table 2.

Table 2. Chemıcal Composıtıon Of Pyrophyllıte Ore Maın Components

Comp.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2	FeO	SO_3	P_2O_5	kr.H_2O	BaO	LOI
Cont.,%	64.15	15.92	1.57	6.65	1.06	0.31	0.64	<0.167	0.37	<0.02	0.18	5.18	<0.01	9.46

Also, pH value was identified in all the samples. In two samples of pyrophyllite ore in distilled water, (10g in 100ml, after the solution settlement of t=4h) pH value of the solution was identified at 9.00 for both samples. The measuring was made using the pH meter CONSORT C830P, measuring pH value to two decimal points.

In the pit and micronised pyrophyllite ore sample, rough and hygroscopic moisture were identified using the standard identification methods, whereby the measured rough moisture of the starting sample was Hg=0.60 %, and hygroscopic moisture of the micronized sample was W_h=0.53 %.

Qualitative mineralogical analysis of the sample was performed using the polarising microscope for released light with qualitative identification of the present materials. The lens zoom was from 3.2 to 50x.

Results of the microscopic analysis of the pyrophyllite pit sample showed that the minerals of quartz and pyrophyllite were predominant in the sample, and that carbonates were less represented. Quartz is mostly clear and somewhat kaolinited. Pyrophyllite is rich in solid inclusions. These inclusions are in the form of opaque, or non-transparent minerals. Feldspat and mica are present in traces. Results of analysed sample microphotography are presented in Figures 2 and 3.

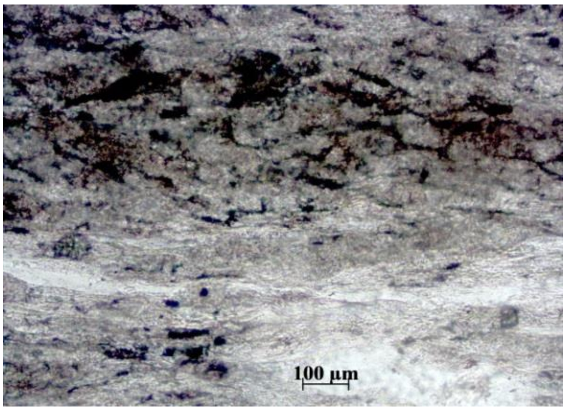

Fıgure 2 Mıcrophotography Of Pıt Pyrophyllıte Sample. Released Lıght, Iı Nıkolı.

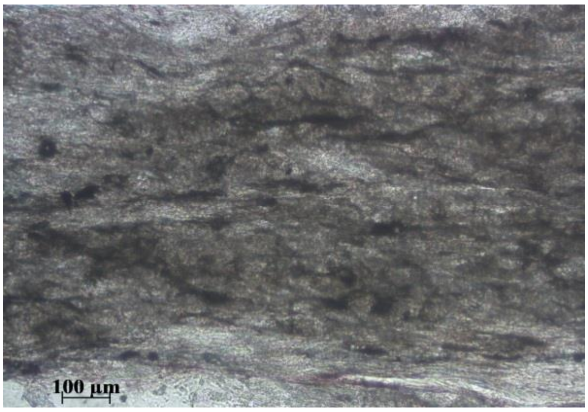

Fıgure 3 Mıcrophotography Of Pıt Pyrophyllıte Sample. Released Lıght, II NIKOLI.

Examination using the ore microscope, by sample size of pyrophyllite samples with size classes of -5.0 + 0.00 mm and -2.00+0.00 mm, showed increase of pyrophyllite content with decrease of size in both samples, and increase in pyrophyllite content in the smallest classes results in decreased silicate content. This is logical since pyrophyllite is much softer (hardness according to Mohs 1-1.5, compared to the predominant silicate minerals and quartz with hardness according to Mohs of 5.5-7).

X-ray diffraction analysis was used for identification and monitoring of sample phase composition. The sample was analysed on PHILIPS X-ray diffractometer, model PW-1710. Mineral composition of the analysed sample was: **quartz, pyrophyllite, calcite, dolomite and kaolinite**. Pyrophyllite is the predominant (≈ 50%), and quartz (≈ 30%) and carbonates (calcite ca10% and dolomite ≤ 5 %) are present to a lesser extent. Kaolinite is present in traces. The difractogram of the analysed sample is presented in Figure 4.

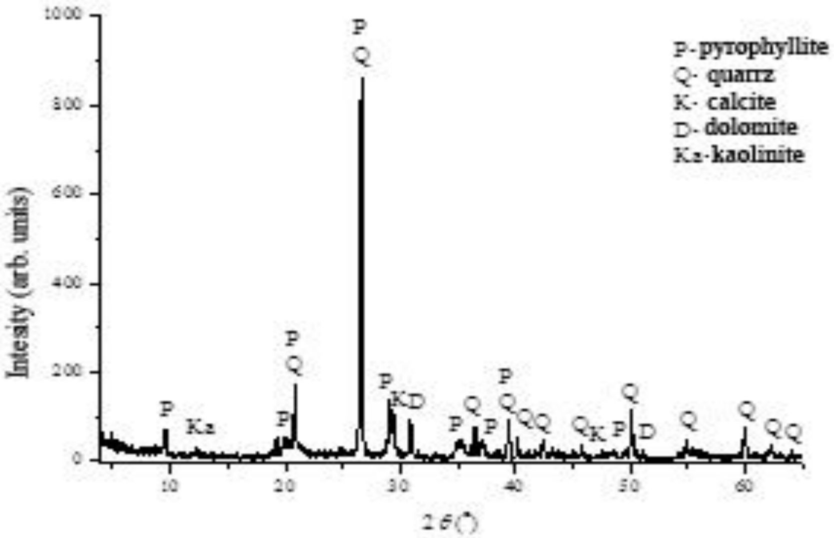

Figure 4 Dıffractogram Of Pyrophyllıte Sample Powder

Semi-quantitative chemical analysis using a scanning electronic microscope (SEM) was performed on the ore preparation of a pyrophyllite pit sample (refer to Figure 5). Based on the chemical analyses performed using a dotted method, it was determined that contaminants or inclusions were: rutile (analyses 3 and 4 of the pyrophyllite pit sample, fluoroapatite (analysis 5 of the pyrophyllite pit sample). Analyses 1 and 2 of the pyrophyllite pit sample (Table 3) show the mean chemical composition obtained by measuring the surface of the analysed sample. Based on these analyses, it may be concluded that the obtained results are in accord with the results of the X-ray analysis.

TABLE 3 CHEMICAL ANALYSES OF PYROPHYLLITE PIT SAMPLE.

	O (%)	Al (%)	Si (%)	Zn (%)	Ca (%)	P (%)	Ti (%)	Fe (%)	S (%)	F (%)
1	57.07	8.56	30.78	-	0.90	-	-	0.34	0.12	-
2	56.62	6.57	33.58	-	1.38	-	-	0.20	-	-
3	60.00	0.86	0.99	-	-	-	38.04	-	-	-
4	44.81	0.82	0.76	-	-	-	53.31	0.30	-	-
5	37.19				32.90	18.64				11.26

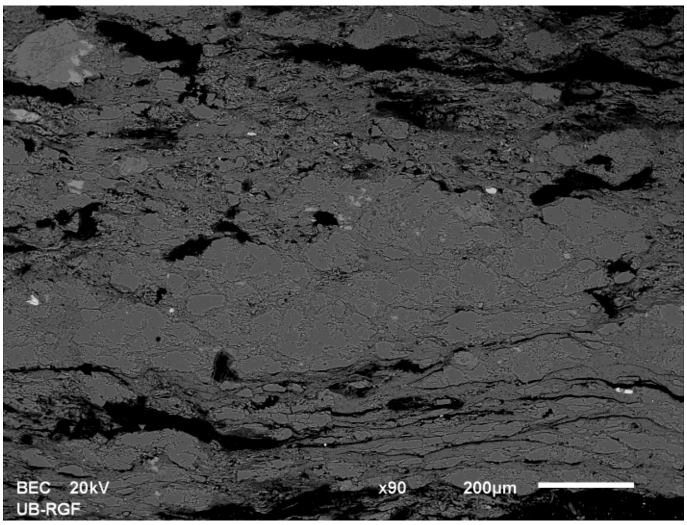

Figure 5: Microphotography of Pyrophyllite Pit

Thermal analyses were performed on the micronized pyrophyllite ore sample, including differential thermal analysis (DTA) and thermal gravimetric analysis (TG). Infrared spectrum was also recorded on the same sample using the infrared spectrometer with Fourier transformation (FTIR), as well as the content of variable cations (KKI).

The results of the thermal analysis of the pyrophyllite sample are presented in Figure 6.

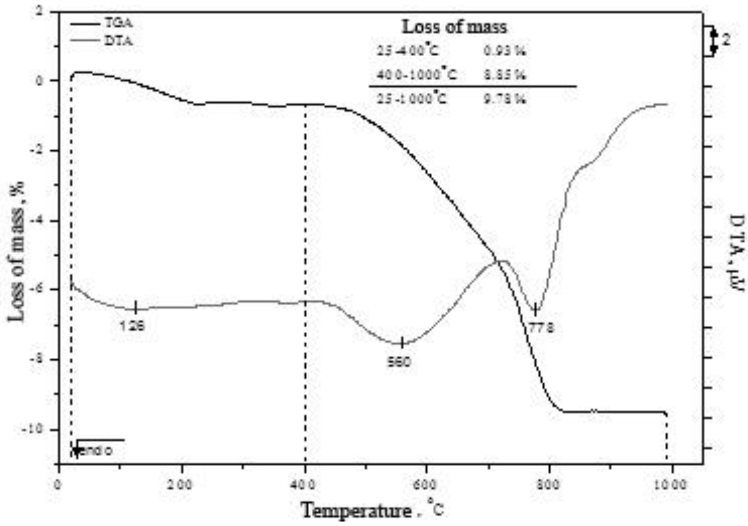

Figure 6 Dta/Tg Analysıs Of Pyrophyllıte Sample

DTA diagram of the analysed pyrophyllite ore sample, within the temperature range of 25-400oC, shows endothermic peak of low intensity with the minimum at 126oC, which originates from the loss of moisture and physically bound water. This process is followed by a drop in mass by 0.93 % at the TG diagram. Within the second temperature range, from 400 to 1000oC, the DTA diagram shows two endothermic peaks, wide endothermic peak with the minimum at 560oC and the peak at 778 oC. Since the sample contains pyrophyllite and kaolinite, and dihydroxylation and loss of structural water with these minerals (endothermic peak on DTA curves) occurs at the temperatures between 530 and 580 oC, it is not possible to determine the mineral from which the peak 560oC originates.

Since the X-ray analysis identified the presence of calcite mineral in the analysed sample, the endothermic peak with the minimum at 778 cm-1 may be attributed to carbonate oxidisation. Due to continuous loss of mass within the temperature range of 400 to 1000oC, it was not possible to determine the exact mass loss originating from each of these two processes. Therefore, the total mass loss was introduced, which is 8.85 % for this temperature range.

The results of FTIR analysis of pyrophyllite sample are presented in Figure 7.

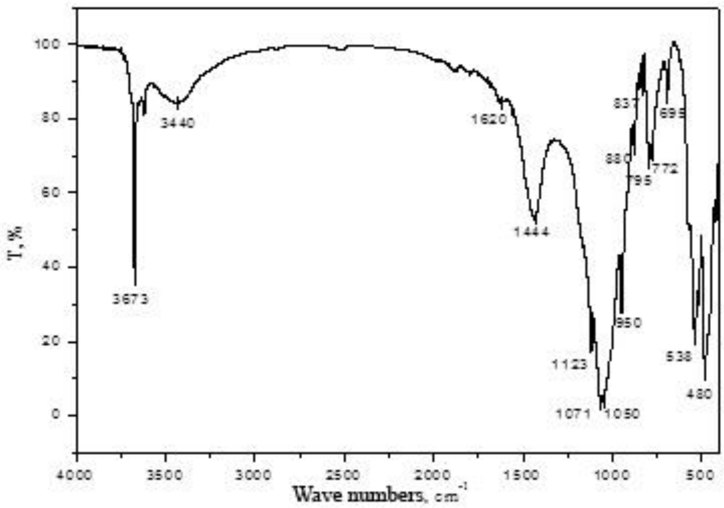

Fıgure 7 Ftır Dıagram Of Pyrophyllıte Sample.

Within the range of the analysed sample, spectral lines characteristic of pyrophyllite are easily identifiable. Sharp and narrow spectral line at 3673 cm-1 originates from a steady vibration of Al2OH group in pyrophyllite. Wide spectral line at 3440, and spectral line of low intensity at 1620 cm-1 originate from the presence of physically absorbed water in the sample and vibrations of OH group. Spectral lines in the range of ~1120 to 950 cm-1 originate from characteristic Si(Al)-O steady vibrations, and strips at 538 and 480 cm-1 originate from bending Si(Al)-O vibrations. Strips at 837 and 880 cm-1 originate from vibrations outside the plane of OH group. In addition to spectral lines characteristic of pyrophyllite, within the spectrum of the analysed sample, spectral lines characteristic of calcite may also be identified. For example, the strip at 1444 cm-1 originates from asymmetrical valence vibration ($\tilde{\nu}$ 3) of CO3 group, which is double degenerated ($\tilde{\nu}$ 3a and $\tilde{\nu}$ 3b), and strip at 695 cm-1 originates from its bending vibration in the plane. Also, within the spectrum of the analysed sample, two spectral lines at 795 and 772 cm-1 are observed, originating from steady vibrations of Si-O group characteristic of quartz.

The results of identification of content and type of variable cations of micronised pyrophyllite sample are presented in Table 4.

Table 4 Results Of Identification Of Variable Cation Content In Pyrophyllite Samples

Sample	Na, meq/100g	K, meq/100g	Ca, meq/100g	Mg, meq/100g	$\Sigma_{cation,}$ meq/100g
1. Pyrophyllite ore	0.56	0.81	52.89	5.43	59.69

The X-ray analysis showed that the following minerals are present in the analysed sample: quartz, kaolinite, calcite and dolomite and pyrophyllite. Since quartz and pyrophyllite do not contain variable cations, the variable cations present in the analysed sample most probably originate from calcite and dolomite (Ca and Mg) and kaolinite (Na, K), [4-5].

4 CONCLUSION

From the experimentally obtained results at the Institute for Technology of Nuclear and other Mineral Raw Materials – ITNMS in Belgrade, in cooperation with the private company "AD HARBI d.o.o."-Sarajevo, and well-known and renowned experts in agriculture, the possibility of use of pyrophyllite in agricultural production was examined for melioration or fertilisation or simultaneously for melioration and fertilisation of agricultural land. This was carried out primarily from the standpoint of creating conditions for the start of production of agricultural products, required under the principles of organic agriculture, which does not exclude their use in conventional agricultural production.

Use pyrophyllite in agricultural production requires prior analysis of its activity in field demonstration experiments and provision of detailed explanations based on such analyses.

The application of pyrophyllite in agricultural production will create a series of positive effects:

- development of conditions for initiation of agricultural plant production (which is also the basis for cattle production) under the principles of organic agriculture, which results in agricultural products of high biological (eco) value, in demand both on our market and on the markets of developed countries;
- there is a realistic possibility for a positive foreign trade balance, which creates conditions for increased financial investments in the agricultural production;
- the country is relieved of import of certain raw materials for the production of mineral fertilisers;
- employment will be increased in the exploitation of pyrophyllite, intended for agricultural production;
- rational use of surfaces of degraded agricultural land in agricultural production will be increased;
- in addition to the production of biologically more valuable (eco) food in compliance with the principles of organic agriculture, complete environmental protection will also be ensured.

The presented advantages of natural pyrophyllites are sufficient reasons for the need to verify their value for the use in agricultural plant production, particularly in compliance with the principles of organic agriculture.

5 ACKNOWLEDGEMENTS

We wish to express our deepest gratitude to AD HARBI, which enabled us to come to the realization of this project.

This research was supported by the Ministry of Education and Science and Technological Development of the Republic of Serbia and carried out under the Projects 34006 and 34013.

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