Characterization of sludge resulting from the extraction and processing of natural stones. focus on a Piedmont case

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Abstract
The problem of the management of sludge resulting from the extraction and processing of ornamental stones is currently one of the key issues for the European Union and for Italy.

The European Community, through Directive 2006/21/EC (Management of waste from extractive industries – Mining waste), Directive 2008/98/EC (Waste Framework) and Directive 1999/31/EC (Landfill Waste), has provided guidelines for its member countries on how to handle wastes, in view of a future sustainable development of the sector, especially as far as critical raw material (CRM) is concerned.

In this context, an analysis of the procedure applied to extract and cut two similar Piedmont silicatic stones into slabs and to manage their sludge has been carried out.

The ornamental stones taken into consideration are Diorite of Traversella and Perosa Stone (Dioritic gneiss). The following procedures were conducted to characterise the stones: petrographic analysis, as well as compressive strength, flexural strength, impact resistance and water absorption tests. This characterization is important to help choose the best available technique to extract and process the stones. The methodologies adopted in two different plants, according to their processed stones, are discussed hereafter. In addition, UPV and Knoop analyses were performed in order to establish the workability class of the two stones, and the classification developed in previous research works was applied.

Subsequently, an analysis was conducted on the sludge resulting from the extraction and processing of blocks used for the production of slabs. A chemical analysis, a particle size analysis, a magnetic separation test, to obtain two fractions (metallic and mineral), and an SEM analysis of the two separated fractions were performed on the two sludges.

This characterization was useful to understand whether the produced sludge could be recovered as by-products. The study will help stone producers to identify the best techniques for the extraction and processing of their stones, while obtaining less waste and less pollution.

Keywords: waste management, diorite processing, sludge characterization, best available technique

1. Introduction

The European Union's primary goal, in terms of waste, is to minimize the level of its hazardousness, to reduce the quantity of waste to be disposed of, and to dispose of what is destined for landfilling safely, while favoring recovery and particularly recycling (Directive 2006/21/EC and Decision No. 1600/2002/EC of the European Parliament and of the Council of 22 July 2002 which laid down the Sixth Community Environment Action Programme[1, 2, 3]). The development of measures on hazardous mining waste and the promotion of the sustainable management of extractive industries, with a view to reducing their environmental impact, also emerge from Decision No. 1600/2002/EC as priority actions.

It is necessary to fix minimum requirements to prevent or reduce, as much as possible, any negative effects on the environment, or on human health, resulting from the management of waste produced by extraction industries.

The main aim of processing industrial and mining waste is to avoid its disposal [4]. Today, no country can afford not to take into consideration the recycling potential of waste produced after the use of primary raw materials. Simply abandoning these wastes, without any additional treatment, because it is cheaper, is no longer an option, as is now aware of the cost to the environment, human health and society. The benefits of dealing with this kind of waste are both of an economic nature , as it can improve the environment and the landscape, and of a social nature, because it creates jobs and better social conditions for the involved communities. For this reason, the appropriate use of such waste should be considered as an integral part of a sustainable development strategy and as a compensation measure for the local communities that are involved. Local authorities can play a key role, in order to find solutions to safeguard the environmental, to treat wastes and to lay down new foundations for sustainable industrial development. An efficient use of resources and better access to raw materials should be the pillars of such a circular economy.

However, in order to do this, it is necessary to create a network of public, private or public-private partnership projects to share the responsibilities concerning future investments, infrastructures and the protection of the environment.

Any treatment of extraction waste should be accompanied by information about the physical characteristics of the waste and its chemical properties , in order to provide sufficient data to the authorities and businesses that will then have to start reprocessing activities or introduce environmental protection programs. In light of what is described in the Legislation, an improvement in cutting techniques, based on the type of cut stone, would help to reduce the amount of

produced mud, but above all to reduce the amount of heavy metals present in this mud. The use of an appropriate cutting technique would ensure increases in the tool life and consequently reduce the working times, the substitution costs and the waste materials. However, this aspect depends directly on the type of stone, and could result in a higher cutting efficiency and consequently a reduction in costs. Many studies have been conducted on the efficiency of the diamond wire cutting of soft rocks; for example, Yilmazkaya and Ozcelik[5]determined the optimum working conditions for a marble sample, in terms of cutting performance parameters, including unit wear and cutting rate, and developed cuttability charts with respect to the cutting performance parameters.
The aim of the present study is to characterize the produced sludge in order to evaluate the best available technique used to extract and process the stones, with the least possible waste and pollution.
Two types of analysis were performed to characterize the stone and the sludges produced during the processing. The following tests were performed on the stone: petrographic analysis, compressive strength, flexural strength, impact resistance, water absorption, UPV and Knoop microhardness.
The following tests were performed on the sludge: chemical analysis, particle size analysis, magnetic separation test to obtain two fractions (metallic and mineral) and SEM analysis.

2. Materials and methods

The materials considered in this research came from two factories of two different districts of the Piedmont Region (Italy). Figure 1 shows the geographical location of the two quarries. The two plants will hereafter be identified with the stone trade names: Diorite of Traversella and Perosa Stone. Information on the type of stone and cutting method used by the two plants is given in Table1.

Figure 1: Piedmont Region districts where the two quarries are located.

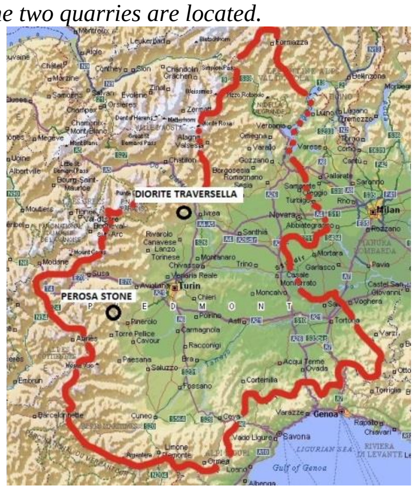
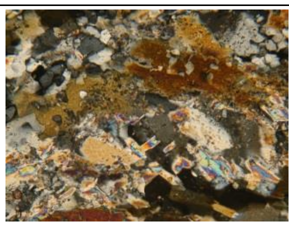


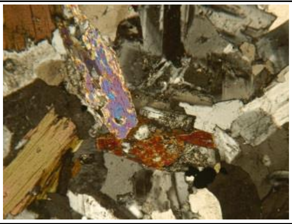
Table 1: list of samples with their acronyms and type of processes used by the two companies

Companies	Type of process	Type of stone processed	Acronym
1	Diamond Saw Blade	Perosa Stone	DBP
2	Diamond Wire	Diorite Traversella	DWD

The two stones have similar mineralogical characteristics, but different structural and textural features, due to the alignment of the minerals. The Perosa Stone, a metamorphic rock, exhibits the usual foliation structure, a feature that is not present in the Diorite Traversella (magmatic rock), which shows a granular isotropic structure. Table 2 shows the macroscopic and microscopic characteristics of the two studied stones.

Table 2:list of the stones with their trade names and a macro-micro description

Trade name of the stone	Macrophoto	Microphoto	Mineralogical composition	Description
Perosa Stone			Quartz 45% Plagioclase 20% Mica 15% Epidote – Zoisite 15% Accessory minerals: chlorite, biotite, zircon 5%	Dioritic gneiss - Metamorphic rock with clear and dark foliation due to lamellar phyllosilicate. Medium-fine grained. Presence of quartz and chlorite veins.

Diorite Traversella			Quartz 15% Plagioclase60% Biotite, Chlorite Opaque and accessory minerals 15%Pyroxene 10%	Magmatic rock, fine grained and light grey coloured diorite of the matrix with small, darker grey grains, isotropic fabric.

Perosa stone blocks are extracted by means of the drilling and blasting technology, while diamond saw blades are used to cut the blocks into slabs. The sludges obtained from stone processing are stored temporarily within the quarry area for about three months, while waiting to be landfilled (*Figure 2*).

Figure 2: Company 1 (Perosa) – cutting methodology: diamond blade (on right) – open-air sludge disposal (on left)

Diorite Traversella is extracted by means of the diamond wire technology. The obtained blocks are cut into slabs with multiwires or monowires (*Figure 3*). The sludge resulting from the processing is canalized into decantation tanks, filtered and stored inside the plant site while waiting to be landfilled.

Figure 3: Company 2 (Diorite Traversella) – cutting technology for the extraction (on left) and for the processing (on right): diamond wire

These two cutting methods are more recent than the traditional gangsaw processing, and, moreover, their tools are worn less during the cutting phase. The diamond blade and the diamond wire methods produce less waste than the gangsaw process. In addition, the produced sludges show a significantly lower concentration of metals (especially of Iron), due to the minor wear of the tools and the absence of metallic grit.

No data is available on the amount of sludge produced by the different plants in Italy, while data on the annual production and number of quarries in Piedmont are available on the Piedmont Region site. *Table 3* shows the amount in tons of production of the two stones, considering the average value from 2011 to 2014.

Table 3: Mean quantity of the produced Diorite Traversella and Perosa stones in tons from 2011 to 2014. (Data source: http://www.regione.piemonte.it/industria/cave/pietre.html[6])

2011 - 2014	
Quarry	**Produced tons (mean value over three years)**
Diorite Traversella	3000
Perosa Stone	120

According to a study by Celik and Sabah[7], the largest amount of waste is produced during the slab cutting phase. The thickness of the cutting tool and the thickness of the obtained slab affect the amount of sludge that is produced. Therefore, the volume of produced sludge was estimated taking into account the tool dimensions and assuming 2 cm as the average slab thickness.

The results of the estimation of the amount of sludge produced in the 2011 to 2014 period are reported in Table 4.

Table 4: Mean value (in tons) of the sludge produced during the Diorite Traversella and Perosa Stone cutting processes from 2011 to 2014 .

2011 – 2014	
Quarry	**Tons of Sludge (mean value over three years)**
Diorite Traversella	600
Perosa Stone	30

These data are important to understand the economic and environmental benefits of recovering sludge from the above-mentioned machining operations rather than sending it for landfilling.

2.1Characterization of the stones

In order to understand and optimize the cutting technology, it is important to analyze the physical and mechanical characteristics of the considered stones as well as their mineralogical aspect. The following tests were carried out on the stone samples: apparent bulk density, absorption coefficient, compression strength, indirect tensile strength, impact resistance (minimum drop height), Knoop microhardness and UPV. All the tests were carried out in accordance with the EN European standards. *Table 5* shows the tests carried out and the relevant reference standards.

Table 5:Tests carried out and the relative reference standards

TESTS	REFERENCE STANDARD
Apparent bulk density	EN 1936:2006
Absorption coefficient	EN 13755:2007
Compressive strength	EN1926:2006
Flexural strength	EN 12372:2006
Impact resistance	UNI EN 14158:2005
Knoopmicrohardness	UNI EN 14205:2004
UPV – Ultrasound pulse velocity	EN 14579:2004

Sakcali[8] and Akcakoca[9] claimed that the quality control of stone blocks is an important aspect of the production process, as it helps to reduce the level of wastage during the production of slabs from blocks. The mechanical and physical characterization of the considered stone is important to choose the best cutting method and the parameters necessary to have a finished product of quality, fuel saving and less produced waste. The UPV and Knoop tests were carried out considering the results of previous studies on the workability of stone cutting with diamond wires [10, 11].The UPV test highlights the mechanical and physical characteristics of a stone, while the Knoop test considers the microhardness of a surface, two features that are closely connected to the workability of a stone. The ultrasonic pulse velocity (UPV) was determined using a PUNDIT–CRO instrument, connected to oscilloscope lap-top software, with 33 kHz conic frequency transducers. Measurements were made indirectly by placing the transmitter transducer on a fixed point and the receiver at progressive distances (each 25 mm: from 25 mm to 175 mm) on the same specimen surface (specimen dimensions 200mm x 200mm x 20 mm). A water-based gel was applied to the transducers in order to prevent air from entering and to improve contact between the transducers and the stone surface.

2.2 Sludge characterization

As previously mentioned, several issues can arise related to the waste originating from the production of ornamental stones. At a national and European level, interest is focused on new technologies, but also on strategies to reduce consumption, reduce costs and time, and to reduce CO2 emissions in an end-of-waste approach. For this purpose, the present research has characterized the sludge resulting from the cutting of slab blocks in order to establish suitable destinations for the new by-products.

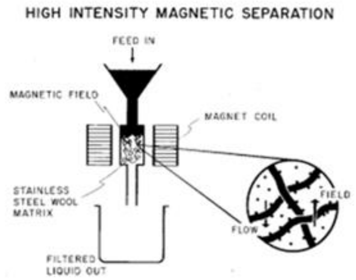
The following analyses were carried out on the sludge: granulometric analysis, chemical analysis on solids and eluates (leaching test), wet magnetic separation and SEM-EDS.

The *particle size distribution* was performed in wet conditions, using two sieves (0.038mm, 0.106mm), in order to obtain three classes. This analysis is necessary to understand which particle size class presents the highest concentration of metals and to choose the best separation method.

Magnetic wet separation was performed to separate the metallic fraction with magnetic characteristics from the mineral fraction of the sludge. This type of separation allows those metals that are part of the CRMs to be separated and identified. Their recovery and re-use is an important aspect for a circular economy (EU pillars).

A Kolm-type high gradient magnetic separator, manufactured by Eriez-magnetics, was used for this purpose. The device is schematically shown in *Figure 4*.

Figure 4: Kolm-type high gradient magnetic separator.(From John A. Oberteuffer, 1974[12]).



Devices of this type are useful for the concentration or removal of weakly magnetic fine grained particles.
The device operates in discontinuous mode. In order to separate the two fractions, it is necessary to perform several steps for both fractions (magnetic and amagnetic).
The operative magnetic, competing, and interparticle forces determine the performance of the separator. These forces depend on:
- the nature of the feed that has to be separated, that is, its size and physical properties;
- the character type of separation device: design, variable parameters (magnetic field, process rate).

The magnetic force in a separator may be maximized by matching the magnetic field gradient to the particle size.
In the present case, the coils and the iron magnet circuits are used to magnetize a ferromagnetic structure whose field gradients attract the magnetic particles.
The *chemical analysis* and leaching test were conducted in accordance with Italian Ministerial Decree 186/2006, which provides two reference tables that show the concentration limits of metals in sludge.
Leaching tests, for the characterization of the eluate, were carried out in accordance with Art. 9 and Annex 3 of D.M. 05_02_1998.
Scanning Electron Microscopy (SEM) is a QUANTA INSPECT 200 LV FEI device with ECAX GENESIS energy dispersive X-ray spectroscopy (EDS), and a SUTW detector. This analysis, which produces only a quality evaluation, was carried out to evaluate the composition of the two fractions after separation.

3. Results and discussion

The mineralogical and petrographic characteristics of the two considered stones show differences on the percentage composition of some minerals, but the main feature that distinguishes them is their schistosity. The Perosa Stone presents phyllosilicate, arranged in almost parallel layers, and foliation planes, while the Diorite Traversella is a magmatic rock which shows a granular isotropic structure. These differences affect the mechanical properties of the rocks. It can be observed, from the results of the performed tests, how the compression, flexural strength and impact resistance values differ for the two stones. The compression resistance is greater for Diorite Traversella, which is more compact, while the flexural strength of the Perosa Stone is greater, due to its foliation planes, which increase its elastic modulus. The results of the rock characterization tests are given in *Table 6*.

Table 6: Results of the tests carried out to characterize the stones

TESTS	***measurement unit***	***DIORITE TRAVERSELLA***	***PEROSA STONE***
Apparent bulk density	kg/m^3	2814	2756
Absorption coefficient	%	0.36	0.32
Compressive strength	MPa	215	122
Flexural strength	MPa	21.1	29.0
Impact resistance	cm	71.3	85.0

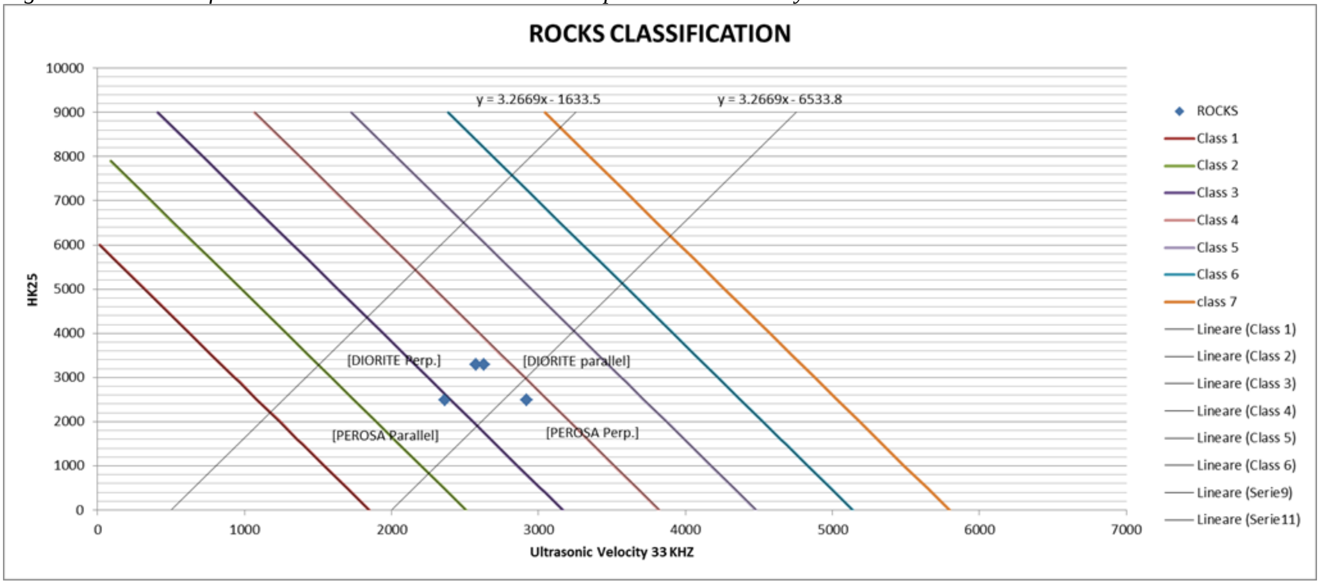
The UPV test was performed both parallel and perpendicular to the foliation planes to verify the differences in the values obtained for the two stones. Knoop Microhardness was evaluated at 40 points per sample, and the mean value was estimated to be HK25 (corresponding to values of 25% below the quartile cumulative frequency). HK25 was chosen, on the basis of the results of a previous study [10, 11], as a value to compare the UPV values. The results of the UPV and Knopp measurements are shown in Table 7. It can be observed, in Table 7, that the Diorite Traversella has UPV values that are practically equal in the two directions (isotropic behavior), while the Perosa Stone has different UPV values, depending on the direction in which the measurement is made.

Table 7: Results of the UPV and Knoop Microhardness tests

Trade name of the stone	***UPV // m/s***	***UPV ⊥ m/s***	***Knoop HK25***
Diorite Traversella	2631	2576	3295
Perosa Stone	2366	2918	2482

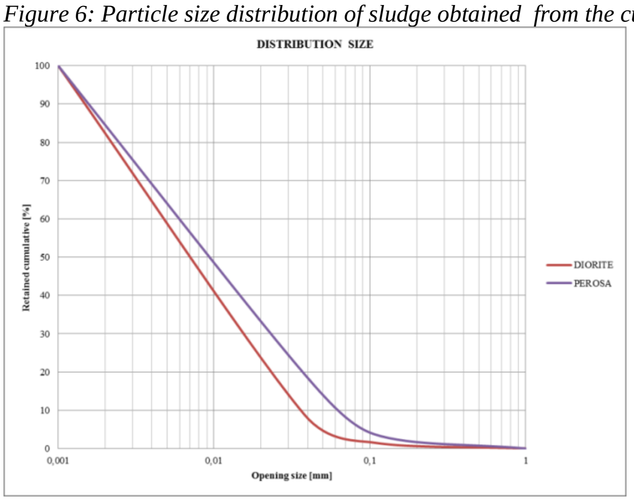
The obtained values were introduced into the classification chart for the workability of stones proposed by Zichella et al [10]. In order to predict stone workability, seven classes were drawn along the correlation line between UPV and HK25, from 1 (easier to cut rocks) to 7 (harder to cut rocks). As can be seen in *Figure 5*, Diorite Traversella always falls into class 3, while the Perosa Stone passes from Class 2 to Class 3 on the basis of the cutting direction with respect to its schistosity.

Figure 5: Rock classification with UPV and HK25 – based on previous research by Zichella et al 2017.



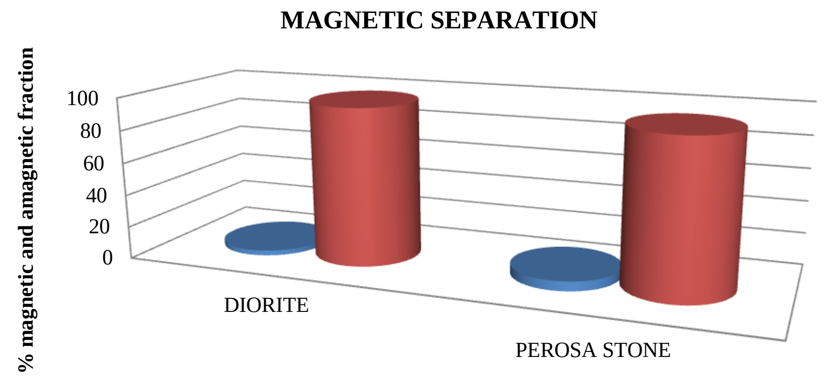
After the characterization of the stones, the sawing sludge was characterized. The obtained granulometric distribution classified the sludge as clayey silt, that is, very fine material (*Figure 6*).

Figure 6: Particle size distribution of sludge obtained from the cutting process



However, the Perosa Stone sludge was found to have a greater grain size than the Diorite Traversella sludge. This aspect is not only due to the petrographic features, but also to the cutting method that is used. In fact, the Perosa Stone plant uses the diamond blade method , while the Diorite Traversella plant uses the diamond wire method, The diamond blade method produces slightly coarser waste than the diamond wire one, due to the dimension of the tools.
As the purpose of this work was to obtain two by-products that could be used in other production processes, it was decided to proceed with a wet magnetic separation that separated most of the metal fraction from the rock fraction.
The results of the magnetic separation are reported in *Figure 7*.

Figure 7: Magnetic separation results obtained using the Kolm-type high gradient magnetic separator.



	DIORITE	PEROSA STONE
% Magnetic fraction	3.1	5.9
% Amagnetic fraction	96.9	94.1

The results of the magnetic separation show that Diorite Traversella has a smaller percentage of magnetic material than Perosa Stone. This is due to the cutting method that is used, since the diamond blade has larger tool diameters than the diamond wire, and therefore produces more waste.
The chemical composition of the solid material is shown in *Table 8* and that of the sludge tested in the leaching test is shown in *Table 9*. The presence or absence of certain metals depends on the composition of the cutting tools, which may contain nickel or cobalt or even both.

Table 8: Chemical analysis on the solid material in an unaltered state, in accordance with Italian D.M. 04/05/2006 no. 186.

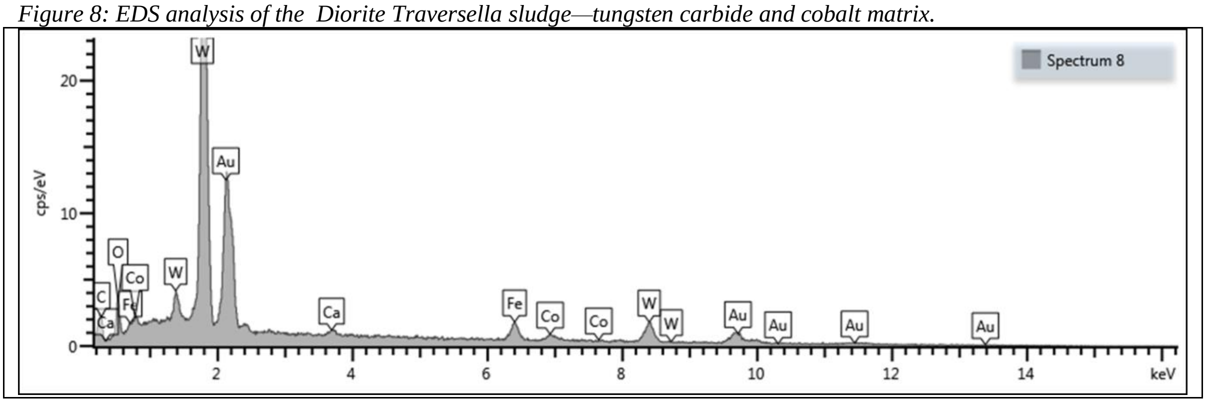
TRADE NAME OF THE STONE	Cr	Fe	Co	Zn	Ni	Cu	Mo	Sn	W
	mg/kg	%	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
DIORITE TRAVERSELLA	18,21	2,75	26,15	24,82	/	41,89	13,15	8,67	33,23
PEROSA STONE	37,75	2,41	26,74	25,04	0,32	38,66	14,36	7,21	24,42

Table 9: Leaching test on the eluate material, in accordance with Italian D.M. 05/02/1998.

TRADE NAME OF THE STONE	Al (ppb)	V (ppb)	Cr (ppb)	Mn (ppb)	Fe (ppb)	Co (ppb)	Ni (ppb)	Cu (ppb)	Zn (ppb)	Ga (ppb)	As (ppb)	Rb (ppb)	Sr (ppb)	Cd (ppb)	Ba (ppb)	Pb (ppb)
DIORITE TRAVERSELLA	89.27	3.12	0.12	17.44	3.28	1.41	0.00	22.47	0.44	2.90	8.28	5.23	102.70	0.00	12.87	0.00
PEROSA STONE	122.60	2.51	0.00	29.40	9.33	1.01	0.00	17.96	0.73	1.05	15.47	8.82	46.58	0.00	4.41	0.00

A SEM EDS analysis was performed on the magnetic fraction, as a qualitative check of the elements that constitute the sludge. *Figures 8 and 9* show the peaks and the related identification photos of the metals.

Figure 8: EDS analysis of the Diorite Traversella sludge—tungsten carbide and cobalt matrix.



Element	Wt%	Wt% Sigma
C	14.05	0.74
O	11.19	0.45
Ca	0.68	0.11
Fe	6.32	0.28
Co	2.74	0.30
W	65.02	0.73
Total:	100.00	

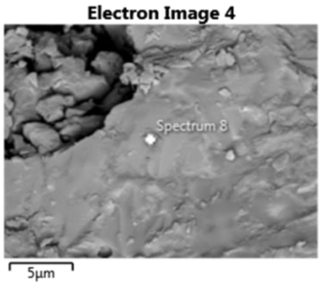
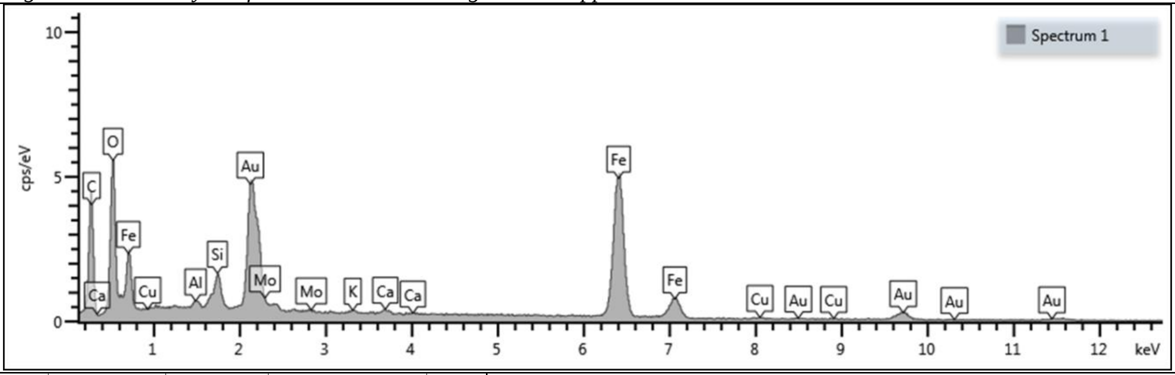
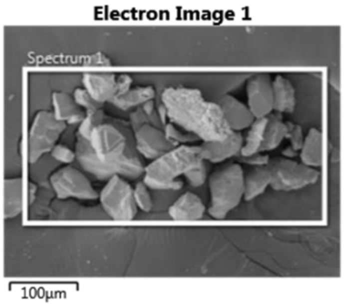


Figure 9: EDS analysis of the Perosa Stone sludge—iron copper and molibdeno.



Element	Wt%	Wt% Sigma
C	39.66	0.53
O	22.60	0.31
Al	0.28	0.03
Si	1.00	0.04
K	0.15	0.03
Ca	0.25	0.04
Fe	34.97	0.36
Cu	0.00	0.00
Mo	1.07	0.19
Total:	100.00	



Two by-products were obtained after the three-step separation: a magnetic fraction and an amagnetic fraction. The amagnetic fraction was cleansed of the magnetic fraction, while the magnetic fraction was still dirty from the rock fraction.
However, the SEM analysis confirmed the results of the chemical analyses carried out to establish the presence of metals in the sludge.

4. Conclusion

The interaction between the mechanical characteristics of a stone and the wear of the tool used to cut it can be observed through the analysis of sludges obtained during processing.
In fact, Perosa Stone, which is characterized by irregular quartzite and chlorite outcrops and schistosity, shows different UPV values, depending on the direction in which the measurement is performed. Graphically, in agreement with the classification of rocks, Diorite Traversella falls into class 3 as far as workability is concerned, while Perosa Stone can either fall into class 2 (cut parallel to foliation planes) or class 3 (cut perpendicularly to the schistosity).
The same metals were obtained from the sludge analysis, at the constituent level, but the diamond blade, used in Perosa Stone plants, produced more waste. In addition, the Perosa Stone, which has more resilient mechanical features and quartz veins than Diorite Traversella, was found to wear the tools more. This aspect emerged in the magnetic separation, where the percentage of magnetic fraction obtained for the Perosa Stone was greater than that of the Diorite Traversella (processed with diamond wire method).
The adoption of the diamond wire method in the Perosa Stone Plant could also reduce the percentage of metals, and in turn reduce the waste. In view of the chemical analyses that were carried out, and in accordance with current legislation, it is possible to state that sludge can be reused. A simple and inexpensive magnetic separation treatment could produce two byproducts that could then be used in other processes. The amagnetic fraction (rocks) could be used in the construction field, as aggregates for concrete, in vegetable soils for the environmental recovery of compromised sites [13], in stone bricks and in lightweight concrete, while the magnetic fraction could be reused in electrical circuits.

The main recovery problem is at a regulatory level. Indeed, there is no indication of what type of treatment is under normal industrial practice. Directive 2006/21/EC defines a treatment as: “A process or combination of mechanical, physical, biological, thermal, or chemical processes on mineral resources, including the exploitation of quarries, the extraction of minerals, the modification of the mineral size, classification, separation and leaching, and the reprocessing of previously discarded material. However, melting and thermal processes (which differ according to the calcination of a limestone) and metallurgical operations are excluded.” Magnetic separation is a simple screening and does not change the actual state of the material, but improves the characteristics for any possible future recovery. Instead of landfill disposal, a systematic recovery of sludges (by means a preventive treatment) is proposed with this method in order to enhance the potential resources that could be reintroduced into production and consumption cycles. Recovery therefore transforms the production system (unidirectional system) into a circular system.

References

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