Geoheritage (2024) 16:105
https://doi.org/10.1007/s12371-024-01013-0

ORIGINAL ARTICLE

Waste From Alwar Quartzite (A Global Heritage Stone From Rajasthan - India) As Secondary Raw Materials for Cementitious Tile Adhesives

Sossio Fabio Graziano[1] · **Paolo Marone**[2] · **Antonio Trinchillo**[3] · **Claudia Di Benedetto**[4] · **Giovanna Montesano**[4] · **Concetta Rispoli**[4] · **Piergiulio Cappelletti**[4,5]

Received: 29 April 2024 / Accepted: 4 September 2024

Abstract

Waste deriving from quarrying operations of natural stone material retains almost all the mineralogical and compositional characteristics of the original material, for such reason this research aimed to test prototypes cementitious tile adhesives made up recycling the Alwar Quartzite waste, used as fine and ultra-fine aggregate. Particle size distribution analysis, along with X-ray diffractometry, X-ray fluorescence and Scanning Electron Microscopy were carried out to characterize the waste. Experimental research involved the mix-designing of three dough formulations (a regular one [N], a latex added [L] and a fast-setting [R]) tested by using different types of tiles: (i) polished metal plates, (ii) ceramic tiles and (iii) rough natural stone slabs. Fresh prepared doughs were firstly tested for thixotropy achieving high values (ranging 82–93%) and cured for normative requested time after being stuck on a concrete support as reported in European UNI standard regulations. After respective curing time, adhesives technical performances were evaluated by the Pull-Off test obtaining results for Class 1 (N and R) and Class 2 (L) adhesives with high initial tensile adhesive strength. Experimental results carried out in this research proved the possibility to use huge amounts of waste coming from Indian stone industry in cementitious tile adhesives sector without compromising technical performances, proposing itself as an alternative method to landfill disposal for this waste.

Keywords Indian quartzite · Stone waste circular recycling · Cementitious tile adhesives · Sustainability · Pull-off test · Initial adhesive strength

Introduction

Alwar quartzite, considered as dimension stone (ASTM International 2020) (Kaur et al. 2021), for its massive use in Indian cultural heritage and building sector is listed as a global heritage stone by International Union of Geological Sciences (IUGS – 2022). As well as all over the world, even in India, the extensive use of stone materials as building stone, as it is easy to guess, produces, in addition to the finished product, a processing waste that accounts for up to 40–50% of the finished product and has a negative impact on the economy of companies and above all on the environment as it has to be landfill disposed (Indian Stone Industry Statistics and Trends; Gayakwad et al. 2015; Patel et al. 2015). The reuse/recycling of by-products or wastes from quarrying and operations, along with the concern on critical and strategic raw materials (Girtan et al. 2021; European Commission 2023), represent not only a need but also an opportunity to combine proper management of natural

✉ Sossio Fabio Graziano
sgraziano@unina.it

[1] Department of Pharmacy, Federico II University, Via Domenico Montesano, 49, Napoli 80131, Italy

[2] Istituto Internazionale del Marmo, via Cenisio, 49, Milano 20154, Italy

[3] c.so Campano 459, Giugliano in Campania, Napoli 80014, Italy

[4] Department of Earth Sciences, Environment and Resources, Federico II University, Via Vicinale Cupa Cintia, 21, Napoli 80126, Italy

[5] Centro Musei delle Scienze Naturali e Fisiche, Federico II University, Via Mezzocannone, 8, Napoli 80134, Italy

resources and land, focusing on a rational management of rock waste, deriving from cutting, grinding and polishing operations, according to the concepts of circular economy and reuse (de Gennaro et al. 2004; Gennaro et al. 2005, 2007, 2008, 2009; Monteiro et al. 2004, 2005; García et al. 2006; Wang et al. 2009; Dondi et al. 2016; Graziano et al. 2016, 2022; Napolano et al. 2016; Lim et al. 2019; Marras et al. 2020, 2022; Behera et al. 2021; Camana et al. 2021; Chinnu et al. 2021; Marras and Careddu 2021; Molinari et al. 2021; Mpatani et al. 2021; Upadhyay et al. 2021; Zanelli et al. 2021). Wastes from industrial operation on ornamental and building stones are mostly identified by sludges and aggregates of different grain size, which, minus any contamination due to the type of processing or collection mechanism (tanks, filter-presses, sedimentation tanks, etc.), have the same chemical and mineralogical composition as the original material. For this reason, they can therefore, if properly characterized, be used as a secondary raw material for other technological applications aimed at a more conscious use of natural resources and, above all, at reducing environmental impact. Thus, depending on the geological nature of the processed material, some tested applications for waste recovery were focused on building sector as useful final destination as secondary raw materials (Trong-Quyen et al. 2003; de Gennaro et al. 2005; Gennaro et al. 2007, 2008, 2009; Mun et al. 2005; Dondi et al. 2016; Mercurio et al. 2018; Graziano et al. 2022, 2024). Furthermore, some research focused also on other possible sectors of use such as agricultural and animal feed (Papaioannou et al. 2005; Eroglu et al. 2017) or transversal sectors with high added value, such as pharmaceuticals, environmental remediation, etc. (Cappelletti et al. 2017; König et al. 2020; Serati-Nouri et al. 2020; Morante-Carballo et al. 2021).

On the basis of the above considerations, possible recycling of waste from the processing of ornamental rock depends strictly on the appropriate selection of the starting material and, above all, on a correct characterization aimed at knowing its geological nature, the absence of contamination due to the wear and tear of the working tools and, when used as a secondary raw material, the physical and mechanical characteristics of the waste-based finished product. In this regard, Alwar quartzite has never been investigated, so this research paper is aimed at exploiting the peculiar features when used as secondary raw material in mix design for tiles cementitious adhesives with experimental-tested technical features suitable to match requirements for the building sector. In the specific case of cementitious adhesives, the physical characterization required for the preparation of the technical data sheet of the adhesive is the evaluation of the tensile strength by pull-off test, along with the evaluation of the thixotropy of the cement paste. (De Barroso and Cruz 1998; Júlio et al. 2004; Benzarti et al. 2011; Hoła et al. 2015; Szemerey-Kiss and Török 2017; Fazli et al. 2018; Afandi et al. 2023).

Materials and Methods

Geological Background

From the geological point of view, the Alwar group of quartzites come from the Jaipur stone working district and are part of the so-called Delhi Supergroup (DeS), which was formerly the main part of the Aravalli Mountain Range located in the northwestern part of India (Fig. 1). DeS extends over 700 km between Delhi in the north and Ahmadabad in the south and consists of lithologic associations of variable age ranging from Archean to Neoproterozoic (Heron 1953; Roy and Purohit 2015). Its main lithological constituents are identified by the Archean Gneissic Complex, the Paleoproterozoic Aravalli Supergroup, the Mesoproterozoic Delhi Supergroup, and the Vindhyan Supergroup of the Neoproterozoic. (Naqvi and Rogers 1987; Gopalan et al. 1990).

The DeS is composed by different lithotypes such as conglomerates, limestones, quartzites, schists, gneisses, amphibolites and mafic lavas, all undergone polyphase deformation and metamorphism from greenschists to amphibolites with syntectonic granitic activity (McKenzie et al. 2013; Roy and Purohit 2015; Sengupta and Basak 2021).

The Jaipur political district, with the homonym capital of the state of Rajasthan, is in the eastern part of the Indian subcontinent and accounts for about 90% of the stone material produced each year by the indian stone industry, with more than 16 million tons/year of processed material on a global Indian average production of 61 million tons/year. This huge volume of final products accounts for almost 70% of waste production (Indian Stone Industry Statistics and Trends). These values make the Indian stone industry both as one of the major industry leaders since the high demand in the international market, and one of the worldwide most waste-producer (Jalalian et al. 2021).

Waste Materials

Waste materials were collected from sieving and filter-pressing operations on quartzite and identified respectively as (i) waste sand (WS) sample, deriving from the squaring and handling operations of dimension stone and (ii) waste powder (WP) sample coming from the cutting and polishing operations collected by the filter-press used to recycle water from the processing plant.

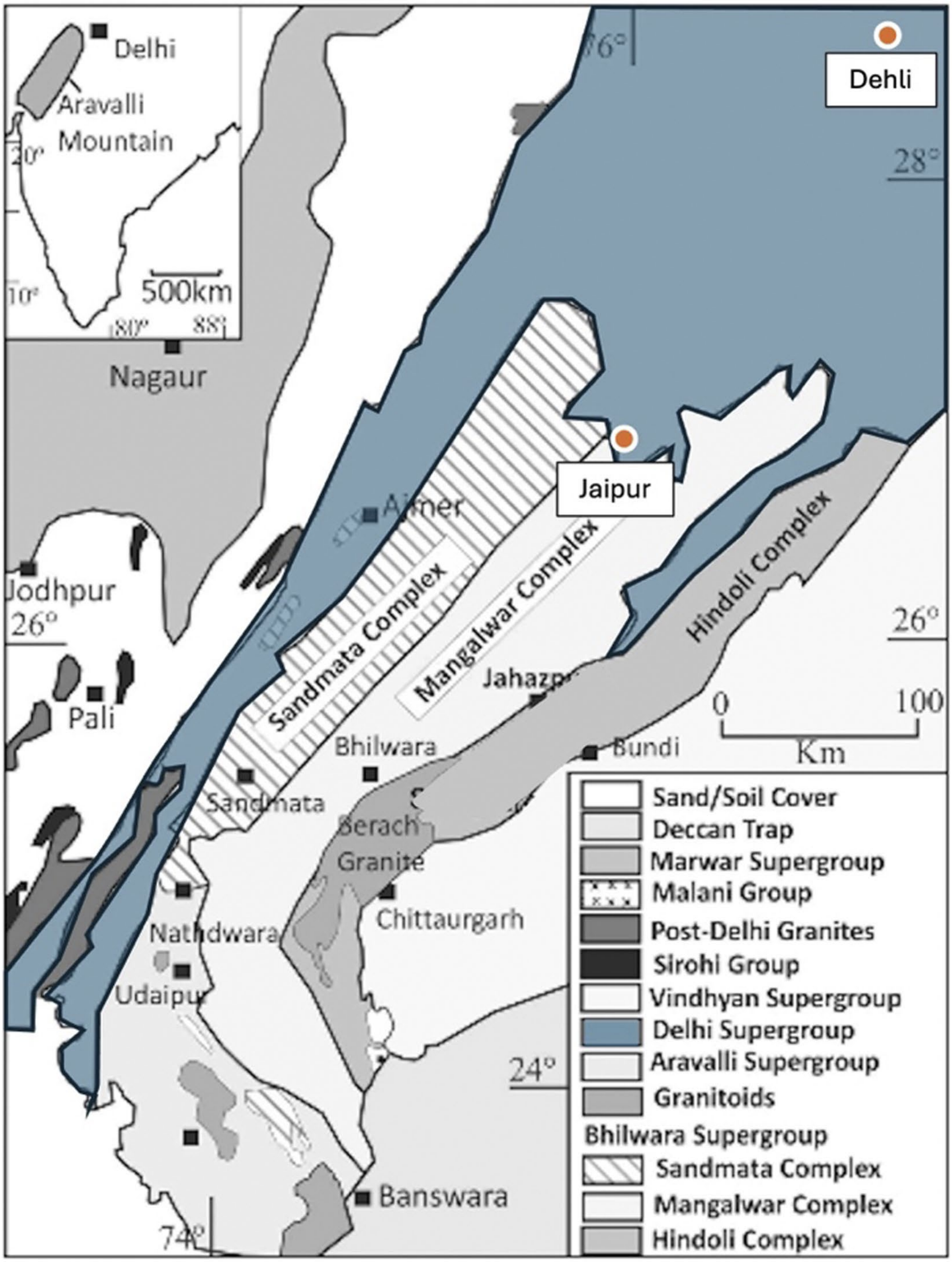


Fig. 1 Geological sketch map (modifed after Sengupta and Basak 2021)

Along with waste materials, other products currently marketed as building adhesives ingredients were used in this study (Fig. 2; Table 1). These products are:

– compound of additives for cementitious tiles adhesives, composed of: (a) Pentamix APL, a powder additive that can reduce the film thickness in cementitious adhesives and retard their formation, and also extending the open time of the adhesive and increasing the tensile strength (Pentamix APL); (b) Pentaresin P3, a water redispersible and highly alkali-resistant resin powder which improves adhesion, impact resistance and protects colors (Pentaresin P3); (c) Penta EC 4119, a medium-viscosity modified cellulose ether suitable for cement-based adhesives

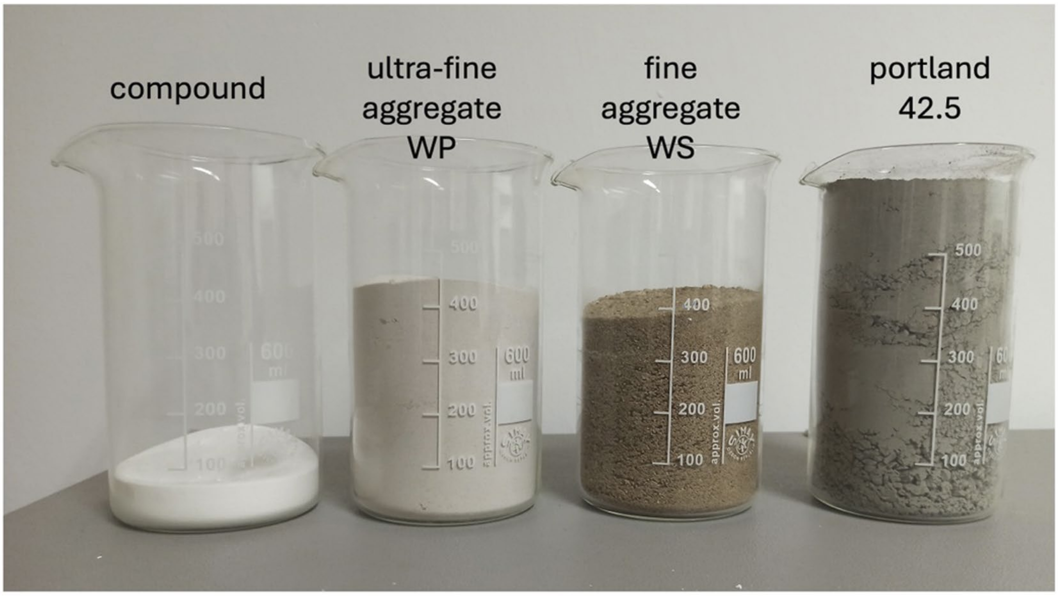


Fig. 2 Waste samples and products currently marketed as building adhesives ingredients

Table 1 Marketed building adhesives ingredients

Component	Name	Property
Compound	Pentamix APL	Penta
	Pentaresin P3	Penta
	Penta EC 4119	Penta
	PentaMix R30	Penta
Cement	Portland CEM I-42.5	Italcementi
Latex additive	Isolastic	Mapei
Fast-setting additive	Penta Rapid CR1001	Penta

and glues which increases the viscosity, water retention, mechanical strengths and thixotropy of mixtures (Penta EC 4119); (d) PentaMix R30, a wetting prepolymer that improves the application characteristics of powder and emulsion resins in cement mixtures by allowing dosage reduction (PentaMix R30);

- Portland cement, CEM I-42.5 (UNI EN 197-1 2011);
- Latex additive (Mapei Isolastic).
- Fast setting additive (PentaRapid CR1001).
- Regular water, conditioned to laboratory temperature.

Methods

Particle size Analysis

The role of aggregates in the mix design is a function of their particle size class, so the particle size analysis of waste samples was investigated by means of the quantitative determination of the distribution of particle sizes, following the indications reported in the in the reference standard (ASTM International 2002). Particles size larger than 75 µm (retained on the No. 200 sieve) were separated by sieving, while the distribution of particle sizes smaller than 75 µm was determined by sedimentation.

Scanning Electron Microscopy - EDS Microanalyses

Microstructural investigations were performed by means of a Field Emission Scanning Electron Microscope equipped with an Energy Dispersive Spectrometer (FESEM/EDS; Zeiss Merlin VP Compact coupled with Oxford Instruments Microanalysis Unit; both used for observations and spot analyses. Data sets were obtained using a INCA X-stream pulse processor with the following operative conditions: 15-kV primary beam voltage, 50–100 A filament current, variable spot size, from 30,000 to 200,000× magnification, 20 mm working distance, and 50 s real-time counting. Data were achieved by means of INCA Energy software 5.05 (XPP array and pulse pile-up corrections).

Mineralogical Characterization

Mineralogical analyses were carried out by means of X-ray powder diffraction (XRPD) using a Panalytical X'Pert Pro diffractometer, equipped with a RTMS X'Celerator detector with Cu-Kα radiation, operating at 40 kV and 40 mA. Sample powders were mixed with a 20% Buelher α-alumina as internal standard and micronized by using a Retsch XRD-Mill McCrone in order to obtain a particle size < 10 µm, a condition that, as reported in literature, allows to obviate several problems when acquiring X-ray spectra (particle statistics, primary extinction, micro-absorption and, especially for feldspathic-type phases, preferential orientation phenomena) (Chipera and Bish 1995, 2002). Samples were acquired between 5 and 70 °2θ, with a step interval of 0.017 °2θ and a time per step of 120 s. Mineral phases were identified by the Panalytical Highscore Plus 3.0e software and PDF-2/ICSD mineral databases (International Crystal Structure Database-ICSD 2012). Quantitative analyses were performed by RIR-Rietveld combined methods, using Topas software (version 5.0, Bruker, Germany) (Rietveld 1969; Bish and Howard 1988; Bish and Post 1993).

Chemical Analyses

Chemical analyses were performed using an Axios Panalytical X-ray fluorescence (XRF) spectrometer, equipped with six analyzer crystals, three primary collimators and two detectors.

Sample powders were mixed with polyvinyl alcohol and poured in aluminum cups, above a layer of boric acid H_3BO_3, to be sure that infinite thickness was achieved; a pressure of 20 ton/cm^2 was applied for 20 s using a hydraulic press to obtain pressed powder pellets suitable for XRF analysis. Analytical percentage uncertainties are 1–2% relative (Cucciniello et al. 2017). The weight Loss on Ignition (L.o.I.), determined by gravimetric techniques, was evaluated by firing at 1000 °C powders previously dried at 110 °C overnight (ASTM International 2021).

Mix Design and Characterization

Three different recipes were tested to simulate the normal working formulations reported in standards for building adhesives (Table 2) (UNI EN 12004-1 2017 Adhesives for ceramic tiles - Part 1: Requirements, assessment and verification of constancy of performance, classification and marking).

Fresh doughs were mechanical stirred by an Ika Werk mod. RW18 by adding, the fine aggregate, the ultra-fine one, the compound and finally the Portland cement in succession and without water. Mixing was then operated for about 30 min on a dry basis. Then the first half of the water was added for 15 min, and the remaining half for other 15 min (UNI EN 12004-1 2017).

Table 2 Mix design for lab experimental simulation

Mix design	Unit	*N*	L	*R*
Compound for cementitious adhesives	kg/100 kg	2.15	2.15	2.15
Portland cement 42.5		37.00	37.00	37.00
WS		34.00	34.00	34.00
WP		26.85	26.85	26.85
Elasticising additive (L)	%	-	6.00	-
Fast-setting additive (R)		-	-	5.00
H_2O	lt	20.00	20.00	20.00
Sample preparation		electric mixer	electric mixer	electric mixer
Curing time		28 days	28 days	6 h
Test tile		metal, ceramic, stone	metal, ceramic, stone	metal, ceramic, stone

The three mix-designs were respectively reported as: Normal (N), Latex (L) and Rapid (R) to identify a normal recipe, another one with the addition of Latex (mix L), which is usually added to improve gripping performance and a fast-setting one (mix R) for faster gripping performance.

The N mix was so assumed as the reference recipe and then integrated with the elasticizing additive (Latex - L) and the fast-setting additive (Rapid - R) in quantity of 6%/100kg and 5%/100kg respectively. Three formulations were so designed and tested to meet the requirements defined by standards for C1 and C2 classes adhesives characterized by an initial tensile adhesion strength and a high initial tensile adhesion respectively ≥ 0.5 N/mm^2 and ≥ 1 N/mm^2. Standard classes for R were marked by the letter F and consider physical features evaluated in not more than 6 h instead of 28 days (UNI EN 12004-1 2017; UNI EN 12004-2 2017).

Fresh doughs rheology was tested for determining the thixotropy of the mass by applying the mixture to two steel plates. The thickness of the coating was so evaluated both on horizontal and vertical disposed plates after 24 h and reported as a percentage in relation to those of the horizontal disposed plate (UNI EN 13062 2004).

The experimental laboratory simulation was also extended to the type of tile used. In fact, three types were used to simulate different grip scenarios: an unpolished natural stone tile (high grip scenario), a ceramic commercial tile (common grip scenario) and a steel tile (low grip scenario). After a curing period of 6 h for R and 28 days for N and L, Pull-Off tests were performed by using a Controls digital removable device mod. 58-C0215 fully complying with the standards requirements. For each dough formulation 10 test tiles of each kind were tested for a total of 90 Pull-off measurements (UNI EN 12004-2 2017).

Results and Discussion

Particle size for sample WP was already provided by the supplier as <0.063 mm, sample WS was sieved and formerly identified as “medium sorted sand” with uniformity, curvature, and sorting coefficients respectively of 6-1.5 and 1.9 (Fig. 3).

This experimental classification allows the possibility to use, from the physical point of view, of both waste samples as suitable ingredients for the mix design of the cementitious tiles adhesives (UNI EN 8520-1 2005; UNI EN 12004-1 2017). Fine and ultra-fine aggregates, i.e. with

Fig. 3 Particle size distribution of WS sample

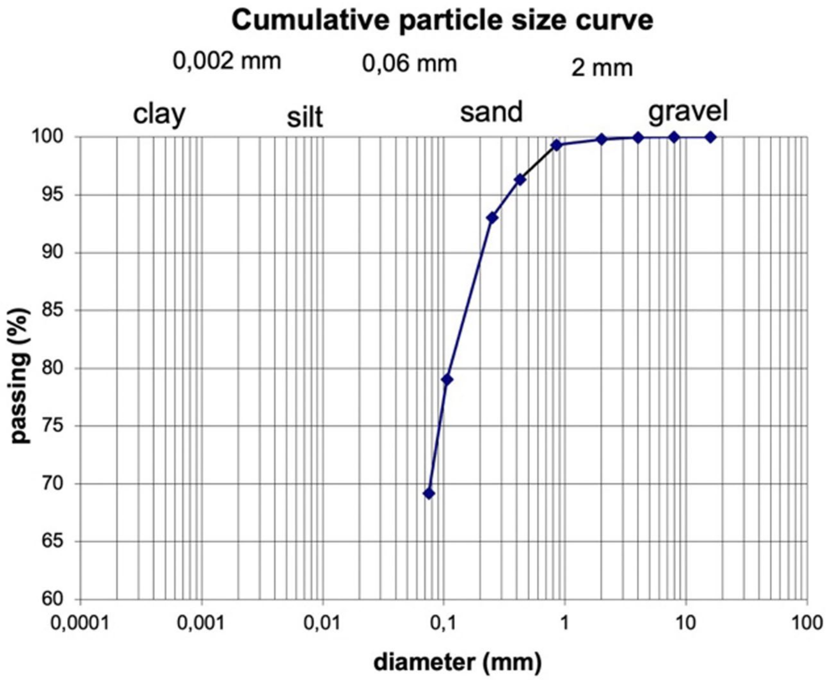


Table 3 Mineralogical and chemical composition (Tr: traces)

	Unit	WP	WS
quartz	wt%	91	84
mica		tr	2
zircon		tr	tr
calcite			2
K-feldspar			1
amorphous		7	10
SiO_2	wt%	91.0	81.7
TiO_2		0.5	0.3
Al_2O_3		3.0	5.4
Fe_2O_3		1.3	1.4
MnO		0.1	0.1
MgO		0.1	0.4
CaO		0.3	3.2
Na_2O		0.2	1.0
K_2O		1.0	2.4
P_2O_5		0.1	0.1
Loss on Ignition	%	2	4

a grain size of less than 75 µm (WP) and between 75 µm and 2 mm (WS), respectively, play the temper role by actively preventing the shrinkage of the fresh mix after the mixing water evaporation.

The mineralogical composition consists of predominantly quartz for both waste samples along with mica and zircon. Small differences can be identified in the higher presence of mica, along with calcite and K-feldspar only for WS sample (Table 2). The chemical analysis is consistent with the mineralogy and with the petrography of the rock (Kaur et al. 2021) reporting high Al, Ca and K values for sample WS (Table 3).

Despite the same precursor, i.e. the processed quartzite, small differences in both mineralogical and chemical composition indirectly reflect the mechanical operations on the rock. In fact, although the typical mineralogical composition of the rock is represented by quartz, mica and zircon (Kaur et al. 2021), calcite and k-feldspar can only be observed in the WS sample, probably resulting from the settling point of the cutting machine and certainly from incorrect sorting of the waste exiting the processing line. This evidence highlights a fundamental requirement for the correct reuse of this type of waste, namely the need to differentiate the collection lines upstream by type of operation and type of material processed.

Scanning electron microscopy observations highlighted the main presence of not well-shaped quartz crystals both for WP and WS, indicating the nature of the sample analyzed or reflecting the result of operations on the stone (Fig. 4). EDS investigations basically confirmed the mineralogical and chemical composition and added information on the relationships between the constituents. Unlike from calcite crystals, zircon and K-feldspar were visible and analyzable. This is closely related with the waste collection mechanism. The presence of calcite therefore could be linked to the the confluence of different materials in the waste collection facilities.

Investigated samples were found to be free of metal contamination and for this reason were considered with all intents and purposes as secondary raw materials to be incorporated within cement mixtures, as fine

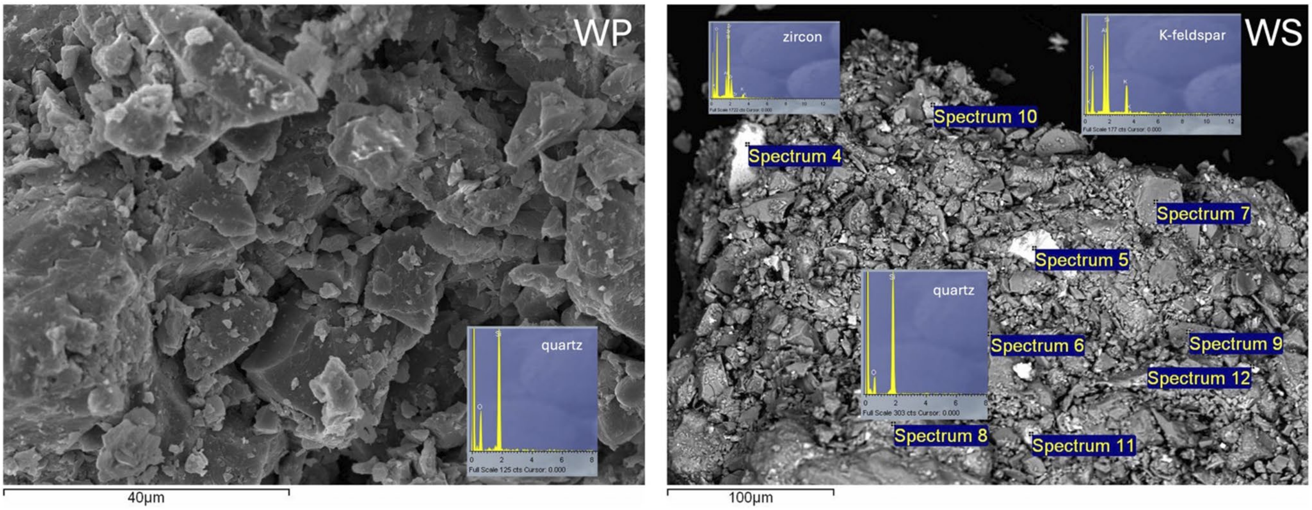


Fig. 4 SEM micrographs and EDS results of WP and WS samples

Table 4 Rheology of fresh doughs (N: regular mix; L: latex added mix; R: fast-setting mix)

	Unit	*N*	L	*R*
Fresh dough thixotropy	%	82	88	93

(WS) and ultra-fine (WP) aggregates(UNI EN 8520-1 2005; Italian Government 2006).

The three mix designs were created following indications of the reference standard with technical additions (Table 1) and rheology of fresh doughs was verified and validated since the thixotropy values were > 80% for all mixes (Table 4).

After curing time (28 days for N and L, 6 h for R – Table 1), dry doughs were analyzed by means of X-ray diffractometry and SEM-EDS and tested by means of Pull-off test to evaluate the behavior of fine and ultra-fine aggregates in the mix.

From mineralogical point of view, all samples were composed of quartz, feldspars, mica and hatrurite. The latter representing a portland related mineral (C3S). Scanning electron investigation, showed that there is no reaction in the mix between the binders and the aggregates, thus acting as the temper and for his reason working as a support for the neoformation of minerals resulting from the setting and hardening reactions of the cement mixture such as C3S and compound-related ones identified by tubular habit and high carbon content (Fig. 5a and b respectively).

The Pull-off test (UNI EN 12004-2 2017), evaluated for mix categories and grip scenarios (cfr. par. 2.5) proved that all the mix designs can be used with profit in regular building applications, this because all mixes can be categorized at least as Class1 (C1-C1F - initial tensile adhesion strength ≥ 0.5 N/mm^2) according to technical performance (UNI EN 12004-1 2017) (Fig. 6).

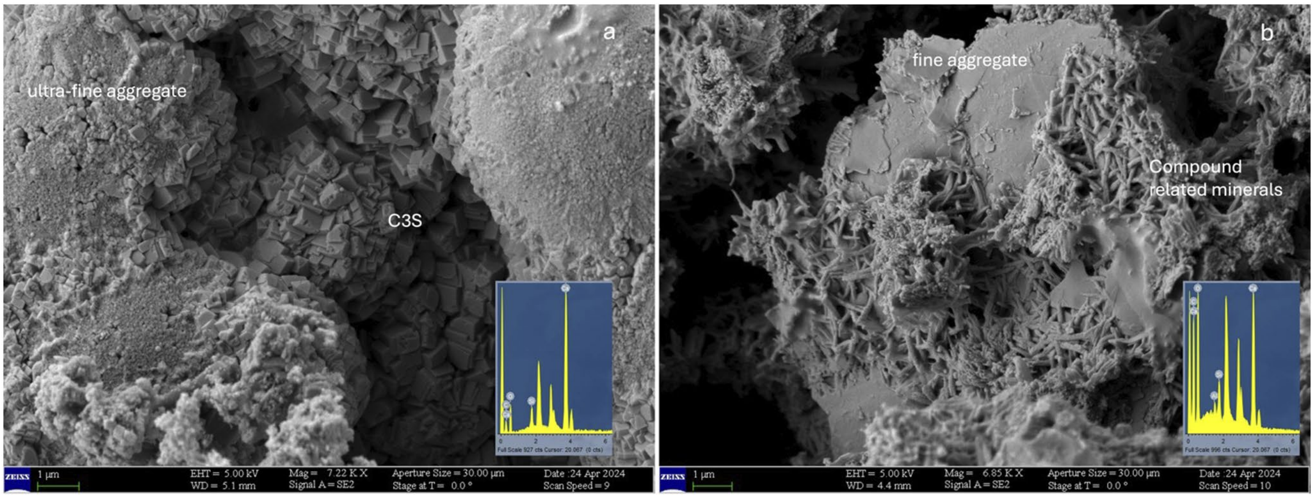
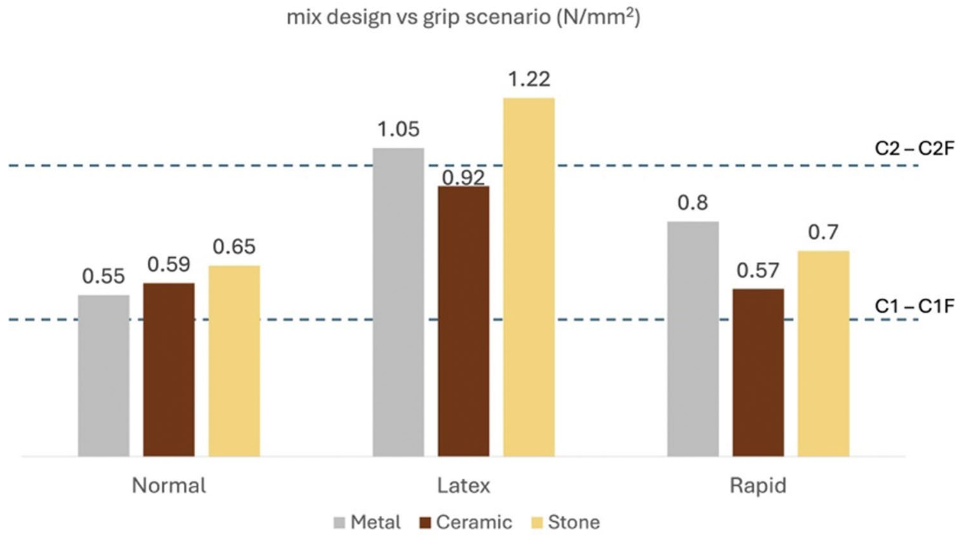


Fig. 5 SEM-EDS micrographs of dry dough after curing time

Fig. 6 Pull-off test experimental results



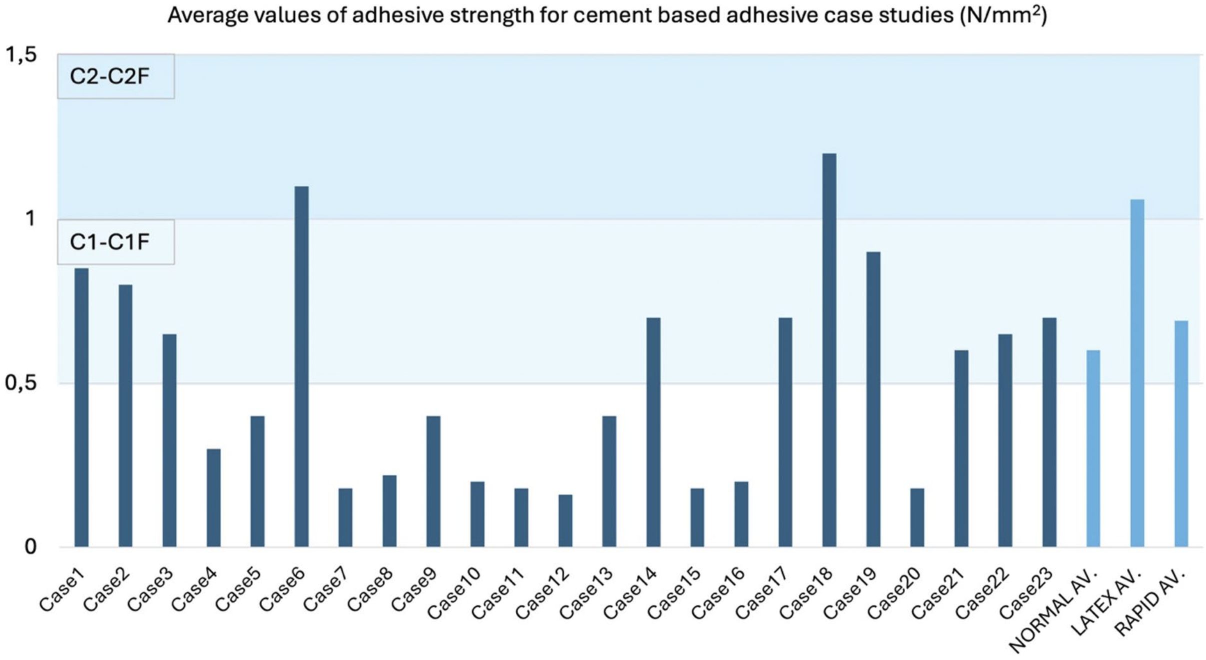


Fig. 7 Comparison between experimental pull-off data and some real cases (Ramos et al. 2012)

From a performance point of view there are no substantial differences between the N mix design and the R, the real improvement refers to the effective setting time (28 days vs. 6 h) with similar initial tensile strength. On another hand, the latex addition (L) determine a substantial improvement in the performance of the cementitious adhesive which can be categorized as Class 2 (≥ 1 N/mm^2) for high initial tensile adhesion strength (UNI EN 12004-1 2017).

By comparing the average results obtained with these experimental compounds with the data of a statistically high number of real cases (Fig. 7), all three compounds tested in this study have a technical behaviour superior to the majority of the samples reported in the literature (performance below class 1) and, for compound L (latex added), even superior to 90% of the average values of real cases of compounds made with products regularly marketed and used in practice. (Ramos et al. 2012).

Conclusions

Arwal quartzite represents a Global Heritage Stone that has been used as the main masonry material in various monuments and buildings in and around Delhi and North India. This geomaterial is is currently extensively quarried and used in numerous heritage buildings and monuments with an indirect production of huge amounts of waste coming from quarrying and processing operations.

This research verified the possibility to use this waste in innovative formulations for cementitious tiles adhesives as fine and ultra-fine aggregates as reported in technical standards.

Three mix-designs were prepared to test a regular (N) recipe, a latex added one (L) and a fast-setting (R) and used to stick three different kinds of tiles, an unpolished natural stone, a commercial tile and a steel tile.

According to technical standards, all formulations for cementitious adhesives tested in this work, can be used with profit in building sector because experimental values for initial and high initial tensile strength fall in the field of class1 (> 0.5 N/mm^2) for N and R and class2 (> 1 N/mm^2) for L.

According to the results reported in this research, this quartzite waste has the same mineralogical and chemical characteristics of the initial rock and can be used, with profit, as a secondary raw material, representing a green alternative to landfill disposal.

Following this approach, finally, it can be argued that waste coming from stone processing can play an important role as technological substitute, enhancing the economies of territories and the industrial development. However, some refinements devoted to the differentiation of processing lines and processed materials are necessary, also from an environmental perspective. Therefore, this study could also spur stakeholders to promote new applied research approaches for a substantial reduction of environmental and social impact of this indian global heritage stone general processing.

Author Contributions All authors: SFG, AT, PM, CDB, GM, CR and PC contributed to the study conception and design. Material preparation, data collection and analysis were performed by SFG, AT and PM. The first draft of the manuscript was written by SFG, and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

Funding Open Access funding provided thanks to the CARE/CRUI agreement with Springer Nature.
Open access funding provided by Università degli Studi di Napoli Federico II within the CRUI-CARE Agreement.

Declarations

Conflict of Interest The author declares no conflict of interest.

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