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RESEARCH

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Hybrid Environmentally Friendly Method for RCA Concrete Quality Improvement

Alieh Mardani[1*], Ngoc Kien Bui[1] and Takafumi Noguchi[1]

Abstract

Research on concrete recycling has led to the discovery of many processes for recycled concrete aggregate (RCA) treatment, each with its pros and cons on quality improvement, resource requirements, or scalability. This study aimed to propose a technique that combines two methods (mechanical grinding and acidic soaking) for mortar removal and improves the quality of RCA so as to realize a zero-waste, highly efficient, few-hours-long single-step process which we call acid milling. A quantitative comparison of aggregate and final concrete quality has been performed between each separate method. The proposed method shows about 57% relative improvement of aggregate quality for water absorption and density, while the concrete experiment shows up to 10% relative improvement for the compressive and flexural strengths compared with the untreated RCA concrete. This study paves the way for an efficient and sustainable concrete recycling process to be applied on a large scale.

Keywords Recycled concrete aggregate, Treatment method, Recycled concrete

1 Introduction

In the context of global awareness of the unsustainability of current production and exploitation models, more and more incentives exist for all industries to recycle discarded items and materials. As the most-used construction material globally, concrete is widely used today because of its properties and availability; thus, improving its life cycle is becoming more and more of interest (Pan et al., 2017; Rahal, 2007; Tripura et al., 2018; Xuan et al., 2016). In this context, recycling concrete as aggregate gathered from demolition materials has attracted considerable attention and efforts to improve it, though so far with mixed results due to a relatively poor-quality material application after processing (Kim et al., 2018; Tavakoli & Soroushian, 1996). The final quality improvement of the concrete made from recycled aggregates depends on the interfacial transition zone (ITZ) between natural aggregates and residual mortar from the previous concrete paste. (Belén et al., 2011; Bui et al., 2018a; Zhou & Chen, 2017). In addition, the micro-holes between recycled aggregates and new mortar after treatment lead to higher porosity, lower strength, and larger crushing value due to changes in geometric characteristics. Besides, the residual mortar decreases the workability and increases the creep and shrinkage of the final concrete (Ajdukiewicz et al., 2012; Lee et al., 2012). These poor properties limit the use of concrete made from recycled coarse aggregates (RCA) to mostly road-based applications (Smith et al., 2008).

Many studies have evaluated a wide range of recycling techniques aimed at either improving or removing the residual mortar attached to aggregates (Bui et al., 2018b, 2019; Shaban et al., 2019; Tsujino et al., 2007). Mechanical treatment is one of the most efficient techniques of recycling based on its environmental footprint and performance (Quattrone et al., 2014). Previous studies showed a 50% improvement in water absorption (Dhir et al., 2003); Wang et al. (Wang et al., 2017) proposed using acetic acid soaking in optimized and minimal concentration as one

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*Correspondence:
Alieh Mardani
alie.mardani@gmail.com
[1] Department of Architecture, Graduate School of Engineering, The University of Tokyo, Tokyo, Japan

of the most promising and sustainable removal processes. The results show significant improvement in RCA quality without undesired residual ions remaining on the aggregates. Moreover, it does not generate new waste and thus can be recognized as an environmentally friendly technique, as the soaking solution itself can be used as mixing water and even enhances the quality of the final concrete. Verma et al. (Verma et al., 2022) proposed another combination of two different techniques in which aggregates were soaked in an acidic solution before using steel ball milling as a mechanical treatment. However, the acid soaking step is not precisely characterized, including the amount of agitation of the aggregates in the solution or whether the aggregates are fully immersed in the solution or not. For example, with no agitation and a shallow layer of soaking solution, the pH value will not be the same in all the solutions, and thus, the action on the aggregates may not be uniform. Besides, aggregates need to be dried between the two steps, which adds potential wastewater and energy consumption. Furthermore, the proposed treatment takes 5 days, which limits its potential applications and requires complex equipment.

The present study proposes to combine acidic soaking and steel milling within a single treatment of only a few hours, which we call acid milling. In this method, an acetic acid solution continuously weakens and attacks the residual attached mortar while the milling gradually removes the mortar mechanically from the RCA; after treatment, the residual solution is filtered to remove floating particles used as mixing water for the new concrete. In this way, the technique does not produce any wastewater, uses only acetic acid as a relatively inexpensive reagent, consumes a small amount of energy (only for the rotation of the milling container), and is as scalable as the capacity of the container, while requiring only a few hours of total treatment time. As the proposed technique involves many phenomena simultaneously, each factor must be studied separately to interpret the results accurately. For this reason, in this study, we prepared six RCA sample groups, each dedicated to the influence of one effect as compared with the untreated RCA: milling, acid soaking without agitation or milling, water milling, acid milling with long rotation phases, and acid milling with short rotation phases. For each group, the aggregate properties were measured before making concrete from them and analyzing the final concrete quality by its compressive strength.

2 Methodology

2.1 Materials

In these experiments, jaw-crushed concrete waste from the origin was sieved between 5 and 10 mm. The initial natural coarse aggregates were also from the same origin with a water absorption value of 0.68% and a surface dry density of 2.67 g.cm^{-3}. The recycled aggregates samples are categorized into 6 groups to be treated differently: untreated aggregates (Untreated), dry-milled aggregates (Dry), wet-milled aggregates (Wet), acid-milled aggregates with 5 min rotation phases (Acid 5), acid-milled aggregates with 10-min rotation phases (Acid 10) and acid soaking only with no agitation or milling (Soaking). For the samples using acid treatment, acetic acid of 99.9% mass concentration is used as a raw reagent before dilution, Portland cement from Taiheiyo company with a density of 3.16 g.cm^{-3}, and river sand with particle size between 0.08 and 2 mm. All experiments were conducted at 20 °C. The particle size distribution of raw materials is shown in Fig. 1.

2.2 Treatment Methods of RCA

Six sample groups of RCAs were selected to study the effect of each phenomenon separately, following the

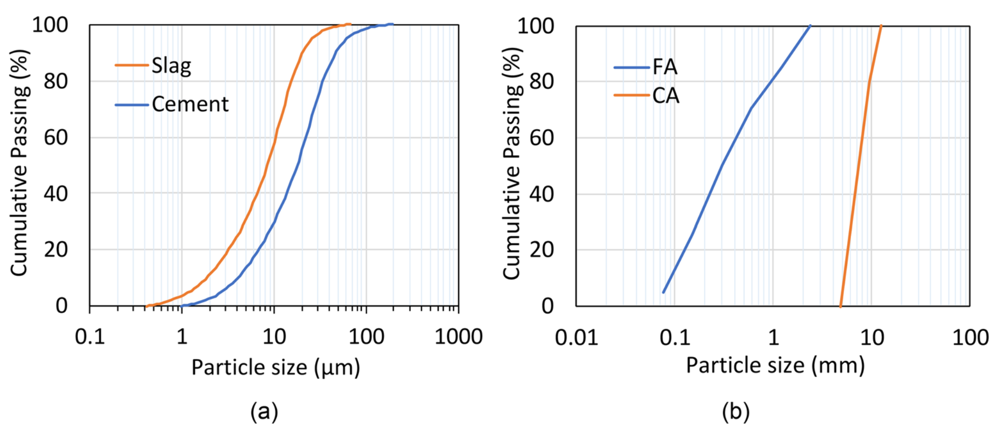

Fig. 1 Particle size distribution of (**a**) cement and slag and (**b**) coarse aggregate (CA) and fine aggregate (FA)

classification of Fig. 2. The "Untreated" group serves as a reference for the recycled aggregates without treatment. The "Dry" group evaluates the efficiency of dry milling itself, while the "Wet" group evaluates the milling in wet conditions with water. Acetic acid with 3% concentration was used for pretreatment of RCA. The choice of acetic acid over others is motivated by three advantages: safer use and transport of the chemical substance, more cost-effective compared with strong acid treatments, and cleaner method overall as it does not introduce undesired ions in the aggregates or concrete. The "Acid soaking" group estimates the efficiency of the acid soaking itself without agitation or milling following (Wang, 2017), while the "Acid 5" and "Acid 10" groups combine acid soaking and milling together with different milling/soaking times. The expectation is that the longer the soaking time, the easier the residual mortar gets removed from the aggregates. Besides, the "NA" group consists of the same natural aggregates as the original ones to scale the improvement of each treatment. For the "Wet", "Soaking", "Acid 5", and "Acid 10" groups, the soaking or milling solution is kept and filtered at the end to get used as a mixing solution to make concrete (Table 1).

2.3 Manufacturing and Characterization of Concrete Specimens of RCA After Treatment

After the treatment stage, RCA samples are used to make concrete. Before casting, as the aggregates are recycled, and the residual mortar can still absorb water, the aggregates were soaked in water for 24 h to be completely saturated so that the proportion of water in the mix design remains the same. The mix design of the concrete specimens was elaborated according to JAS5 industrial Japanese standards, adapted to the aggregates parameters of each group, as shown in Table 2, and set to produce 1 m^3 of concrete. The water/cement ratio for all samples was 50% to achieve medium-strength concrete. In each group that uses wet conditions, the residual solution after aggregate treatment was used as mixing water to verify the effect of solution pH in the final concrete performance. During the treatment phase, the pH of each solution was measured at the beginning and the end after sieving and filtering. The aggregate after the treatment process was used in the concrete mixing procedure, following the JIS standard. Initially, cement was mixed with sand for approximately 15 s until a homogeneous mixture was achieved. Following this step, water with a chemical admixture (specifically, a water-reducing agent from BASF) was added to the mixer and mixed for about 30 s. Next, all material was scraped off from the wall of mixer and then mixed again for 60 s. Subsequently, coarse aggregate was added and mixed for 90 s before being poured into the mold. The mold size of concrete samples is 40 × 40 × 160 mm, with three samples for each group. After casting, all specimens were cured in an air-conditioned curing room at 20 °C for 28 days.

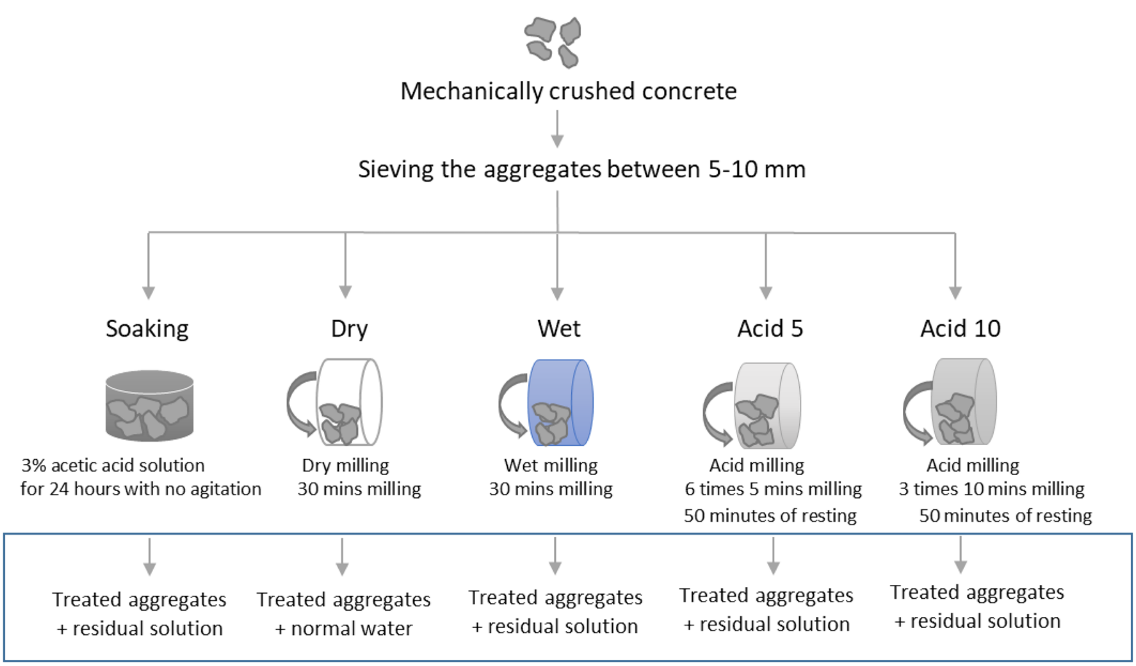

Fig. 2 Sample groups of different RCA treatment methods and their denomination

Table 1 Treatment method of each sample group

NAME	METHOD	CONCRETE MIXING SOLUTION	DESCRIPTION
Untreated	Untreated	Water	Reference, untreated aggregates
Soaking	3% acid soaking for 24h, no agitation	Soaking solution	Soaking without agitation
Dry	Dry milling for 30 min @ 30 rpm	Water	Dry milling
Wet	Milling with water for 30 min @ 30 rpm	Milling solution	Water milling
Acid 5	Milling with 3% acid 6 times 5 min @ 30 rpm spaced with 50 min resting intervals	Milling solution	Short milling with acid
Acid 10	Milling with 3% acid 3 times 10 min @ 30 rpm spaced with 50 min resting intervals	Milling solution	Long milling with acid
NA	Natural aggregates	Water	Reference, raw aggregates

Table 2 Concrete mix design of each sample group based on JIS

		Untreated	Dry	Wet	Soaking	Acid 5	Acid 10	NA
Air content (%)		4.5						
W/C (%)		50						
Ratio cement/slag (%)		60						
Maximum size of CA (mm)		10						
Ratio CA/FA (%)		50						
Water content (kg/m^3)		185						
Extra water (kg/m^3)		72.12	38.39	42.29	54.59	36.27	33.10	5.89
Mass (kg/m^3):	Cement	222.00						
	FA	840.31						
	CA	755.95	806.24	803.00	792.94	817.60	820.52	866.27
	Slag	148.00						
Chemical admixture (%)		3.70						

2.4 Experimental Program

After the aggregate treatment phase, the water absorption, density, and mortar removal of each group are measured to verify the effect of each technique. In the next step, the concrete samples were prepared in 40×40×160 mm molds and cured under water for 28 days. The compressive strength was measured by the universal testing machine with 250 kN axial load capacity according to JIS A 1108, and the flexural strength of each sample was measured following JIS A 1106. SEM images on the surface of aggregate after the treatment process were observed using Hitachi TM-4000.

3 Results and Discussion

3.1 pH of the Treatment Solution

The pH was monitored during the treatment process, and the final pH is indicated in the bar graph in Fig. 3, while the pH of the original solution is indicated as 3.03.

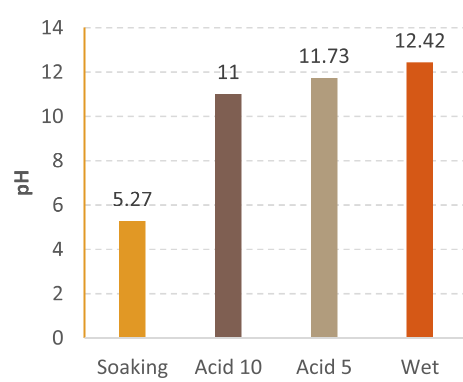

Fig. 3 Final pH in each group of samples with wet conditions, with the initial pH indicated by the dashed red line

In the samples which contained an initially acidic solution with acetic acid, air bubbles were produced due to the same chemical reaction and decreased over time. The final pH in the Soaking group is 5.27, which shows that the solution is still acidic at the end and is expected to cause lower strength in the final concrete (Arunakanthi et al., 2012).

Although the starting solution and its pH are the same between the Soaking, Acid 5, and Acid 10 groups, the final value varies greatly depending on the rotation conditions. This suggests that both the soaking duration and the number of rotations influence the amount of mortar reacting with and dissolving in the solution.

3.2 Characterization of the Treated RCA and Mortar Removal

3.2.1 Mortar Removal

All the techniques used on the samples intend to remove the residual mortar still attached to the RCA. Higher residual mortar in RCA leads to higher water absorption and lower concrete quality; quantifying the amount of mortar removed from the initial RCA is thus one of the main parameters that quantify the expected quality improvement for the final concrete. For this, we use the percent of removed adhered mortar W defined as follows:

$$W = \frac{W_1 - W_2}{W_1} \times 100\%$$

where W_1 is the initial dry mass of RCA and W_2 is the mass of RCA after treatment and oven drying. While this quantity cannot estimate the exact proportion of the residual mortar that has been removed, it enables a relative comparison between different treatments applied on the same initial batch of RCA.

Fig. 4 shows W for the different samples, including the raw experimental data used for the calculation. Both dry and wet milling show similar performance for mortar removal, while acid soaking without any mechanical agitation or milling is significantly less efficient at removing residual mortar than all the other methods at below 6%, which is expected due to the slow diffusion-driven chemical reaction speed. Finally, the last two samples combine wet milling and acid reaction with the same speed and number of rotations, only with the reaction time being different. Both samples show similar mortar removal percentages significantly higher than without agitation or milling.

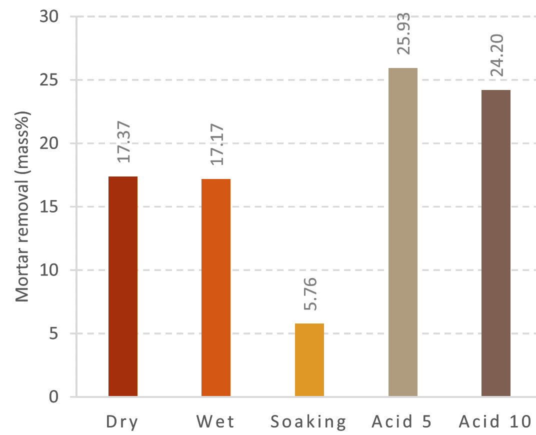

Fig. 4 Mortar removal for each treatment group

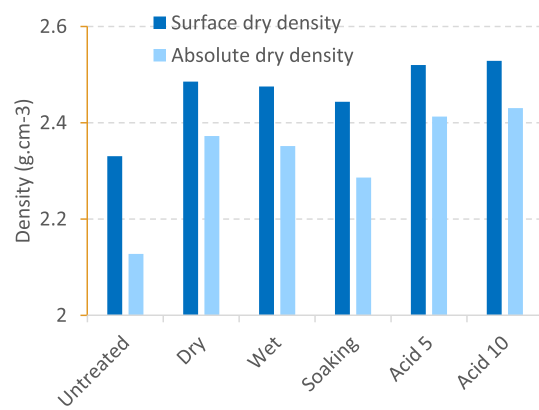

Fig. 5 Surface dry (dark) and absolute dry (light) density data of each experimental group after treatment

3.2.2 Density and Water Absorption

Data for surface dry and absolute dry densities are given in Fig. 5. The trend is similar for both quantities, with acid milling samples showing the highest densities among RCA samples. As residual mortar has a lower density than the aggregates, this is coherent with the results of Fig. 4.

The water absorption is shown in Fig. 6. The data are compatible with the density results, with higher water absorption of untreated RCA being attributed to more residual mortar on the aggregates (Tam et al., 2007).

To explain these results, the recent study of Verma et al. (Verma et al., 2022) provides an interesting comparison: instead of milling with an acidic solution, they use a two-step protocol where aggregates are first soaked in 3% acetic acid solution before being milled in dry conditions. They report slightly lower improvement than the combined technique of the present study, both in terms of water absorption reduction and density variation. Our interpretation is that with the two-step technique, the acidic solution only weakens the external adhered mortar which can then be removed more easily with the milling, whereas the acidic mill-and-wait

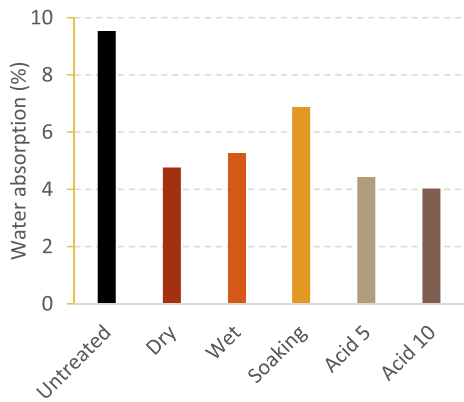

Fig. 6 Water absorption of each experimental group before and after treatment

method continuously weakens the mortar during and between milling intervals.

3.2.3 SEM Images of Treated Aggregates

Each treatment has a different effect on the residual mortar of RCA and the aggregates themselves, resulting in macroscopic (density, water absorption) and microscopic (mortar surface state, ITZ, cracks) changes. While the macroscopic changes are directly related to the amount of remaining residual mortar on the aggregates, the microscopic changes are also expected to influence the properties of the final concrete—even with similar amounts of residual mortar.

Fig. 7 shows a typical surface and interfacial zone of untreated RCA and treated RCA with different methods. For mechanical milling for removing old cement mortar from RCA, as shown in Fig. 7b, c for both conditions, including wet and dry, the large porosity cement mortar was removed; then, they reduced the large gaps in the interfacial zone in RCA and increasing density compared to untreated RCA. Only acid soaking (Fig. 7d) improved the micro-scale interfacial zone and make the RCA denser, but the large pores are still existed in the old cement mortar parts and make cement mortar parts more porous. The proposed combined method (Fig. 7e, f) shows better surface characteristics and a denser interfacial zone between old cement mortar and aggregate in RCA. Large gaps between old cement mortar and aggregate were removed and show only small pores in the interfacial zone in RCA; this established densification of RCA after combined treatment with acid solution and mechanical removal.

3.3 Compressive and Flexural Strength of RAC

The compression results of concrete made with treated and untreated RCA are shown in Fig. 8. The results showed that the mechanical properties of recycled aggregate concrete significantly increased after RCA was treated with the combination method, as shown in sample Acid 5 and Acid 10. These compressive strengths increased by about 10% compared to the untreated RAC. The combined method significantly improved the physical properties and microstructure surface of aggregate, which led to increasing the compressive strength of RAC. In addition, the solution after the combined treatment process with a pH value of around 11, which agrees with the optimal value reported by Bekir Çomak (Çomak, 2016), was used in the concrete mixture. While for the wet and acid milling samples, the improvement in compressive strength is negligible because of the mixing solution, the solution from the treatment process was used in the concrete mixture instead of industrial water for concrete. As these solutions have different compositions and pH compared with industrial water, the concrete quality is expected to vary according to these parameters as well (Çomak, 2016). To decouple the influence of the pH from that of the acetic acid, the concrete made from the wet milling sample uses only the water used during the milling, whose pH value (12.4) is only due to the reaction of the residual mortar with water. The final compressive strength for this concrete is 52.3 MPa, a value lower than with untreated RCA and water. In accordance with Bekir Çomak (Çomak, 2016), the very alkaline pH is responsible for the drop in strength observed here. Because it is mainly due to the high alkali hydroxide concentration in the initial solution does not accelerate OPC hydration, which adversely impacts the nucleation and precipitation of the C–S–H phase (Zajac et al., 2022). This observation aligns with prior research findings that when a solution with a pH of 12 is employed, the resulting compressive strength is lower compared to the use of regular water. Furthermore, the compressive strength exhibits minimal improvement even after a 21 day curing period (Utepov et al., 2022). In addition, within the wet milling method, the surplus of water present in the ITZ also contributes to the decreased strength of the resulting new concrete. Similarly, the pH of the solution from the acid-soaking sample group has a pH of 5.27, a value known to reduce the quality of the final concrete (Arunakanthi et al., 2012).

The same phenomena also were seen in flexural strength of RAC which was indicated in Fig. 9. The combined method improves about 11% compressive strength of RAC compared to that of untreated RCA concrete.

4 Conclusion

In this study, we proposed a new method combining two existing treatments for RCA, acetic acid soaking and steel milling, into a single-step procedure. The results

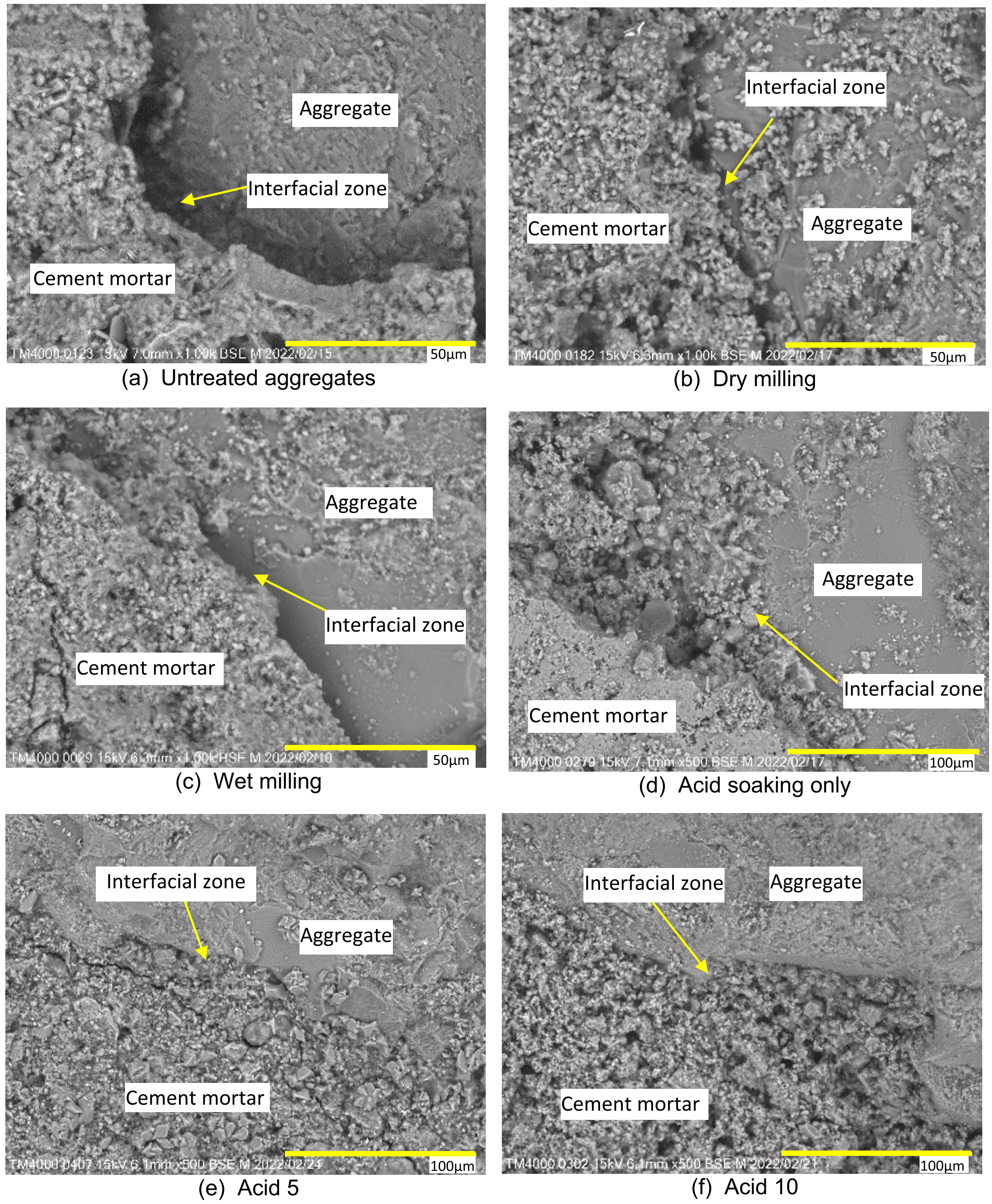

Fig. 7 Typical SEM images of aggregates of different sample groups before and after being treated with different methods: (**a**) untreated aggregates, (**b**) dry milling, (**c**) wet milling, (**d**) acid soaking only, (**e**) acid 5, (**f**) acid 10

obtained in this study demonstrate the feasibility of the proposed methods for improving the mechanical properties of RAC. From the results and discussion, the essential conclusions have been summarized as follows:

– Results show significantly higher mortar removal from recycled concrete aggregate by the proposed combined method than dry milling (25% compared with 17%) and the combined treatment procedure increased the density of RCA compared with solely mechanical or acid treatment.
– The soaking solution after the treatment procedure was used as mixing water for the final concrete, the compressive strength improves by up to 10% compared with untreated aggregate concrete, all the while consuming very limited energy, generating no wastewater (no washing), and needing only a few hours of treatment that can be scaled up. This performance

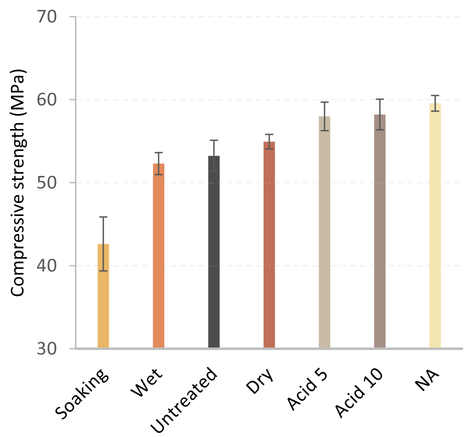

Fig. 8 Compressive strength of each concrete sample

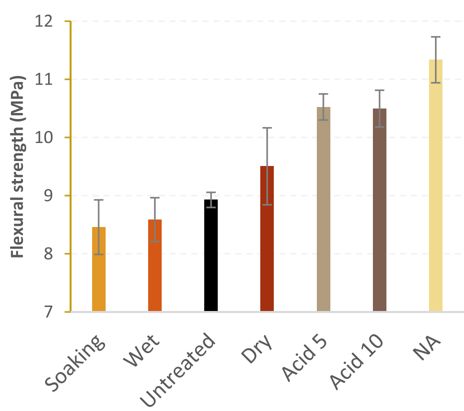

Fig. 9 Flexural strength of each concrete sample

improvement is mainly explained by the uniform and soft acidic attack on the residual mortar combined with the mechanical action of milling, as well as the alkaline pH level for the residual solution after treatment when used as mixing water.

While more research is needed to find the optimal combination between the number of rotations and soaking duration and to confirm these findings with other sources of materials, this method shows promising applicability to the industrial scale thanks to inexpensive raw materials (acetic acid, water), existing machines required and increase in product value. This study paves the way for efficient and up-scale recycling of RCA into high-quality concrete without waste after the treatment process, a big step toward a sustainable solution for valorizing concrete waste.

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Author contributions
AM: data curation, writing—original draft. BNK: writing, review, and editing. TN: supervision and review.

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Availability of data and materials
The authors agree to provide any supporting data upon reasonable request.

Declarations

Ethics approval and consent to participate
Not applicable.

Consent for publication
The authors give their permission to the journal for the publication of the present research findings.

Competing interests
The authors declare no competing interests.

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Publisher's Note

Alieh Mardani is a special researcher at the Department of Architecture at the University of Tokyo, after her PhD thesis obtained at the University of Rome La Sapienza.

Bui Ngoc Kien is a project researcher in the Department of Architecture at The University of Tokyo, and he also serves as a lecturer in the Department of Civil Engineering at Thuyloi University.

Takafumi Noguchi obtained Dr. of Engineering from the University of Tokyo in 1995 and has been serving as a full professor at the same University since 2014. He is a chairman of ISO/TC71/SC8 (Environmental management for concrete and concrete structures). He is a project leader of the NEDO Moonshot Program "C4S Research and Development Project".