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Effect of Recycled Concrete Aggregate Addition on the Asphalt Mixtures Performance: ITZ Area, Microstructure, and Chemical Analysis Perspectives

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Abstract: The importance of environmental consciousness and sustainability is increasing among transportation governing bodies worldwide. Many government bodies are concerned with maximizing the usage of recycled substances in road construction. Therefore, assessing the effect of recycled materials consumption is essential, mainly when designing new 'green' pavement types. The primary objective of this study is to investigate the impact of different treatments on improving the interfacial transition zone (ITZ) of coarse recycled concrete aggregate (CRCA) and its application in asphalt mixes. Such an aim is accomplished by enhancing its physical and mechanical characteristics, as well as its microstructure. The surface morphology, chemical composition, and intermix phases of the ITZ area and calcium silicate hydrate (CSH) compounds for CRCA were evaluated using scanning electron microscopy (SEM), an energy-dispersive X-ray analyzer (EDAX), and X-ray diffraction analysis (XRD). The performance of asphalt mixtures that included treated and untreated CRCA was also examined using different tests. It was found that heat treatment is an effective technique for enhancing the ITZ. However, cracks were seen in the mortar of CRCA when exposed to high temperatures (500 °C), which adversely affects the characteristics of the mortar. Acid treatment appeared to be an effective approach for improving the ITZ area. Nevertheless, the treatment that used acetic acid, a weak acid, was more effective than HCl acid, a strong acid. The outcomes revealed that the ITZ microstructure is significantly enhanced under different treatment types; however, microstructure improvements mainly included increased surface homogeneity and CSH compounds and a reduced Ca/Si ratio. It was also found that the asphalt mixtures with different proportions of untreated CRCA exhibited enhanced resistance to rutting. Furthermore, their tensile strength ratio (TSR) values were above the minimal level requirements. Moreover, the asphalt mixture with 30% CRCA, which was treated with various treatment methods, demonstrated a significant improvement in the mixtures' mechanical properties; therefore, its application is highly successful and an environmentally friendly solution.

Keywords: coarse recycled concrete aggregate (CRCA); interfacial transition zone (ITZ); treatment method; calcium silicate hydrate (CSH); tobermorite; jennite; permanent deformation; thermal cracks; indirect tensile strength (ITS)

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1. Introduction

Over the last fifteen years, there has been a substantial rise in awareness of the need for sustainable development and the protection of non-renewable aggregate resources both in Canada and globally. The rise in energy expenses, along with this heightened awareness, prompted considerable changes in landfill reuse and recycling practices, resulting in a substantial increase in the volume of processed materials. Concrete is the most widely used substance worldwide; billions of tonnes of these materials are extensively spent establishing different structures such as buildings, bridges, dams, and roads. The global production of concrete was estimated annually to range between 6 and 15 billion tonnes, signalling 1 to 2 tonnes per person per year [1–3]. To establish the structures above, enormous quantities of construction and demolition (C&D) waste are generated. More precisely, it was anticipated that the quantity of C&D waste materials generated annually may range from 900 million to 1200 million metric tonnes worldwide [4,5]. Managing these extensive quantities of unusable (waste) materials is becoming a critical issue and an urgent priority at the level of municipalities, cities, and countries. This is due to numerous reasons, including the significant increase in waste materials, the considerable deficiency of enough landfills to dispose of the substances, and the massive increase in transport and waste elimination costs [6]. Thus, the elimination of C&D waste into landfills is an unsustainable management option and an environmentally unfriendly solution because of serious environmental consequences [7].

Solid waste substances comprise a high proportion of wreckage from construction work, which results from C&D works. In general, the definition of C&D is a form of waste materials resulting from different types of construction, renovation, and demolition, including land excavation, structure and building construction, site clearance, demolition work, and roadwork and building renovation [8–10]. According to some estimates, C&D waste consists of 50% concrete waste by weight [11]. Waste concrete may be produced by crushing local C&D waste into smaller chunks. Compared to the fine fraction, the coarse fraction of the produced crushed concrete is separated as the major product and is generally known as the recycled concrete aggregate (RCA) of the crushing process [12]. Various benefits are obtained by recycling and reusing C&D waste. Nevertheless, major advantages can be epitomized in the following points: (1) reduced consumption of natural resources, more precisely, natural aggregates; (2) reduced need for more landfills. In other words, the availability of space in landfills is increased; (3) lower energy consumption and decreased greenhouse gas emissions concerning the carbon footprint of the concrete industry; (4) serious health concerns related to C&D waste disposal are minimized; (5) participation can occur at various levels, including government strategies and industry standards that lead to achieving environmental sustainability [13]. For instance, C&D waste is contributing to global warming. Rising temperatures caused by global warming result in more frequent and severe weather events, such as heat waves, and contribute to the deterioration of air quality [14]. Epidemiological research on heat-wave-related fatalities indicated that a significant proportion of mortality could be linked to increased levels of particulate matter during occurring heat waves. According to some studies in the US, the following health effects have been attributed to rising concentrations of particulate matter in the air: large numbers of premature deaths and hospital admissions due to respiratory disease, significant numbers of asthma attacks, and increased school absences for children because of respiratory-related illnesses such as asthma. The most susceptible individuals to the detrimental impacts of air pollution are vulnerable populations, such as older people, those with cardiovascular or respiratory conditions, young children, and infants [15]. Therefore, recycling the enormous amounts of C&D waste (e.g., RCA material) present in landfills can significantly improve air quality in cities and municipalities, resulting in lowering the health concerns related to C&D waste and its different constituents.

2. Literature Review

Concrete is widely acknowledged as a composite material consisting of many phases, including natural aggregate, cement paste serving as the matrix, and an interfacial transition zone (ITZ) between them [16–22]. Among these phases, the ITZ seems to be completely different from the matrix cement paste and aggregate in terms of structure and mechanical properties. Water film, a $Ca(OH)_2$ crystal layer (denoted CH in terms of cement chemistry), and a porous paste layer are the major components of the ITZ [23,24]. The ITZ region generally stands out for a variety of characteristics, including comparatively lower levels of cement particles, higher percentages of water, and, therefore, elevated porosity levels [25–30]. From the point the view of composite materials, it is agreed that the properties of such materials are highly dependent on its constituents' properties, particularly the connection area. Therefore, the ITZ area significantly impacts the characteristics of concrete [17,22]. Due to the existence of pores and tiny cracks that directly affect strength characteristics, it has been widely reported that the ITZ can be classified as a weak region [18,31–33]. As a result, the ITZ has a key role in concrete performance evaluation, precisely, mechanical properties behaviour [18,34], although it has thin layer; typically ranged from 10 to 50 µm [21]. Therefore, enhancing the microstructure of RCA has been a significant focus for several studies aiming to improve the qualities of concrete [5,21,24]. Nevertheless, different elements significantly influence the ITZ region's microstructure and qualities. These include the kind of aggregate, the aggregate surface conditions, the quantity of cement the aggregate contains, and, most critically, the ratio of water to cement [17].

From the RCA literature, the addition of various materials for ITZ improvement can be described as the main approach for RCA microstructural investigations [18,31–33]. Pozzolanic materials including fly ash and silica fume are extensively used in a successful way for enhancing microstructure. Following their earlier research, [18,19] dealt with utilizing silica fume and cement substances by implementing two-mixing methods to enhance recycled aggregate (RA). In the first technique, known as the "Two-Stage Mixing Approach", silica fume was added in different proportions of RA in the pre-mix. For the second approach, silica fume and quantities of cement substance were added as a percentage of RA in the first stage of the mix. The utilization of silica fume and proportional cement percentages resulted in the RA's weak zones being filled up, leading to an enhanced ITZ, according to the findings of both approaches, and increasing the strength of the concrete. Comparable findings were registered by other researchers who investigated adopting a two-stage mixing technique to improve the characteristics of RCA and RCA microstructures [18,22,35]. The findings of these investigations demonstrated that implementing this technique enhances the ITZ and RCA properties. Employing an alternative technique, specifically triple mixing, demonstrated that the characteristics of ITZ along with RCA were considerably improved in comparison to the previous method, two-stage mixing. The mentioned method focuses on utilizing some of the pozzolanic materials like silica fume and fly ash as coated substances of the RCA surface. The scanning electron microscopy (SEM) images showed that the particles of the pozzolanic substance that coated the RCA particles are capable of consuming CH particles present in the pores and on the surface of the attached mortar. This resulted in the generation of new hydrated compounds, which ultimately led to enhancing the microstructure of the ITZ and increasing the strength of the RCA [21]. The impact of several types of RCA on the microstructure of the ITZ area was evaluated [31]. The adjusted concrete blend used two varieties of RCA, normal and high strength, while preserving the same water-to-cement ratio of 0.5 for both mixtures. The RCA types used in this study were known for having a high-water absorption ratio of 8.82% and 6.77% for normal and high strength concrete, respectively. The SEM images showed that the ITZ of the normal strength concrete-containing mixture is predominantly composed of loosely packed and porous hydrates. In contrast, the ITZ of the high strength concrete-containing mixture consists primarily of dense hydrates. The impact of residual mortar on the characteristics of self-compacting concrete made with untreated and treated RCA was investigated [34]. Different aggregate surface treatments were used in order to maximize RCA use, namely the "two-stage mixing procedure", "pre-soaking in HCl solution", "water glass dispersion", and "cement-silica fume slurry". The study looked at replacing natural aggregate with treated and untreated coarse RCA in 100% of cases. The findings of the SEM pictures revealed that the ITZs that were generated in the treated RCA by the use of the two-stage mixing technique, strong acid (HCl), and water glass had a microstructure that is significantly denser, more interconnected, and less porous in comparison to the untreated RCA. On the contrary, the new ITZ had a porous microstructure that showed a lower level of adhesion due to using the cement–silica fume treatment procedure. The ITZ improved significantly with the two-stage mixing technique, which includes applying a cement slurry layer to the RCA's surface to fill in microcracks and voids.

From the previous discussion, it can be stated that a great portion of the knowledge on ITZ improvement is mainly based on only one approach. The previous microstructure studies of RCA have extensively highlighted the influence of the addition of different materials, including fly ash and silica fume, on the enhancement of the ITZ region, resulting in a noticeable gap in this field. Additionally, though the literature related to the improving physical characteristics of RCA revealed the use of various methods and techniques (the methods and procedures were comprehensively reviewed in the authors' previous papers [6,36]), none of the aforementioned research has investigated the impact of treatment techniques on the ITZ zone. More precisely, the relation between improving physical properties and enhancing the ITZ region under the influence of treatment method application. Hence, an obvious lack of knowledge is found in this regard. Therefore, this research mainly aims to examine the influence of various treatment techniques in improving the ITZ of RCA and its application in asphalt mixtures by improving its physical, mechanical properties, and microstructure. However, this study focuses entirely on coarse recycled concrete aggregate (CRCA), which is a part of RCA material. Initially, various treatments were implemented in a variety of settings to assess each treatment individually, followed by an evaluation of the integration of multiple treatment approaches. Prior to implementing the mechanical method, heat treatment and pre-soaking techniques were employed to evaluate different CRCA characteristics. The influence of these treatment approaches on ITZ improvement in terms of the calcium silicate hydrate (CSH) phase and its main compounds, namely tobermorite and jennite, was also examined. Various aspects, such as surface morphology, chemical composition, and intermix phases of ITZs for CRCA under different treatment types and conditions, were investigated to evaluate the ITZ enhancement. In addition, various performance tests were evaluated for mixtures that included untreated and treated CRCA with different treatments. Such tests included permanent deformation (rutting), moisture sensitivity, and low-temperature cracking resistance (thermal cracks).

3. Results and Discussion

3.1. ITZ Microstrcture

To investigate the surface morphology and texture, different series of SEM micrographs of the ITZ between the adhered mortar and aggregate surface for both treated and untreated CRCA were analyzed utilizing the images shown in Figures 1–3. Figure 1 presents the SEM micrograph of the ITZ between the aggregate surface and the adhered mortar for untreated CRCA. From the captured image, it was found that the surface aggregate morphology is clearly uniform, and its surface structure is very different from that of the adhered mortar paste. In contrast, it was seen that the morphology of the adhered mortar paste is rough. It has a highly porous structure and is irregular, which results in surface heterogeneity. The presence of many voids and various particles lacking specific shapes and sizes was seen under high magnification. This can explain the lower density and higher water absorption of untreated CRCA. Laboratory tests found that untreated CRCA has a relatively low density and high water absorption. Another critical point was that there is a microcrack interface between aggregate and adhered mortar. Contrary to earlier studies, it was revealed that the microcrack width within the ITZ microstructure is not consistent. Notably, the maximum observed microcrack width reached 129.7 μm, representing a range of 2–3 times larger than the typical ITZ thickness in concrete and recycled concrete. The size of microcracks contributes to heightened porosity within the ITZ, causing a reduction in interface bonding and consequently resulting in a weaker region compared to the surfaces of both aggregate and mortar. Therefore, such an outcome proves valuable for comprehending the characteristics of the ITZ, emphasizing the importance of investigating the strength of recycled concrete.

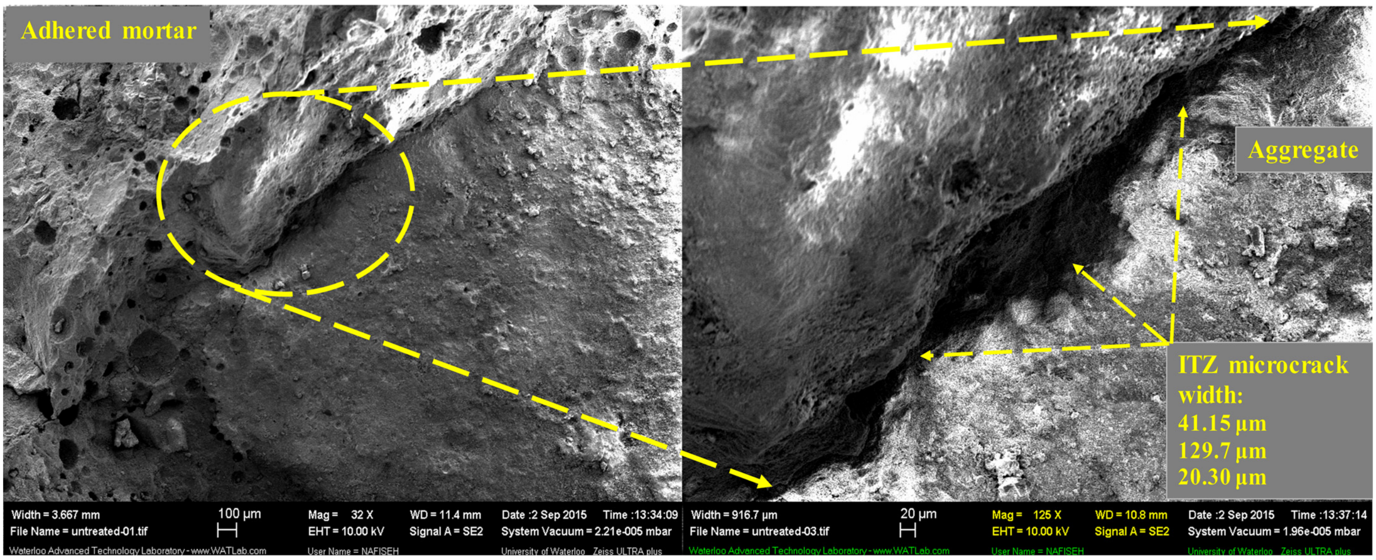

Figure 1. ITZ microstructure of untreated CRCA.

The SEM micrographs of the ITZ between the aggregate surface and the adhered mortar for heated CRCA at temperatures of 250 °C, 350 °C, and 500 °C are shown in Figure 2A–C, respectively. The overall analysis of the surface morphologies for treated CRCA with heat treatment demonstrated that a modification of the ITZ is successfully achieved at various temperatures. However, some important microstructure characteristics due to the temperature variations of the heat treatment can be discussed as follows. Due to accumulated particles being densely packed together, a dense ITZ was clearly visible for treated CRCA at temperatures of 250 °C and 350 °C, resulting in high heterogeneity and a rough ITZ region. This could be attributed mainly to a significant accumulation of CSH particles caused by various reactions. The conversion of CH crystals that accumulate on the surface of the CRCA to CSH particles is due to the completion of the service life and concrete component hydration for the CRCA. This seems to be the main source of CSH particle production. This is essentially accomplished through pozzolanic action, wherein CH crystals are converted into CSH particles via the pozzolanic reaction (Equation (3)). This approach has been widely approved by numerous investigations and has become commonly agreed upon; therefore, pozzolanic materials, including fly ash and silica fume, being added is viewed as a highly successful method for microstructure enhancement, especially the ITZ of RCA. Because CSH crystals are smaller in size than CH crystals, the accumulated CSH particles can easily fill pores and microcracks within the ITZ, leading to improvement and densification of the ITZ.

$$2Ca_3SiO_5 + 6H_2O \rightarrow 3CaO{\cdot}2SiO_2{\cdot}3H_2O + 3Ca(OH)_2 \quad (1)$$

$$2Ca_2SiO_4 + 4H_2O \rightarrow 3CaO{\cdot}2SiO_2{\cdot}3H_2O + Ca(OH)_2 \quad (2)$$

$$Ca(OH)_2 + H_4SiO_4 \rightarrow Ca^{2+} + H_2SiO_4^{2-} + 2H_2O \rightarrow CaH_2SiO_4{\cdot}2H_2O \quad (3)$$

where Ca_2SiO_4 and Ca_3SiO_5 are dicalcium silicate (Belite) and tricalcium silicate (Alite) compounds (cement components); $Ca(OH)_2$ is Portlandite, which is also known as the CH compound; $CaO{\cdot}2SiO_2{\cdot}3H_2O$ and $CaH_2SiO_4{\cdot}2H_2O$ are different calcium silicate hydrate (CSH) compounds; and H_4SiO_4 is silicic acid, which is also known as the SH compound.

It is worth noting that there is a noticeable decrease in the level of roughness of the ITZ surface, which depends on the treatment's temperature. Roughness vanished broadly from the ITZ, and the surface differed from other moderate treatment temperatures as a result of exposure to the elevated temperature of heat treatment at 500 °C, as can be seen in Figure 2C. This means that CSH particles are highly influenced by heat treatment at high temperatures. Additionally, it is interesting to note a crack with 30–40 µm length and 3–6 µm width found in the mortar surface perpendicular to the ITZ, indicating a negative impact on heat treatment at high temperatures on the mortar properties. This observation aligns with the results reported in an earlier investigation [35]. Though heat treatment at different temperatures is registered as a successful approach for improving the ITZ region, various pores, tiny cracks, and ITZ microcracks for treatment at 250 °C and diverse pores on the mortar surface for treatment at 350 °C are still noticeable on the CRCA surface.

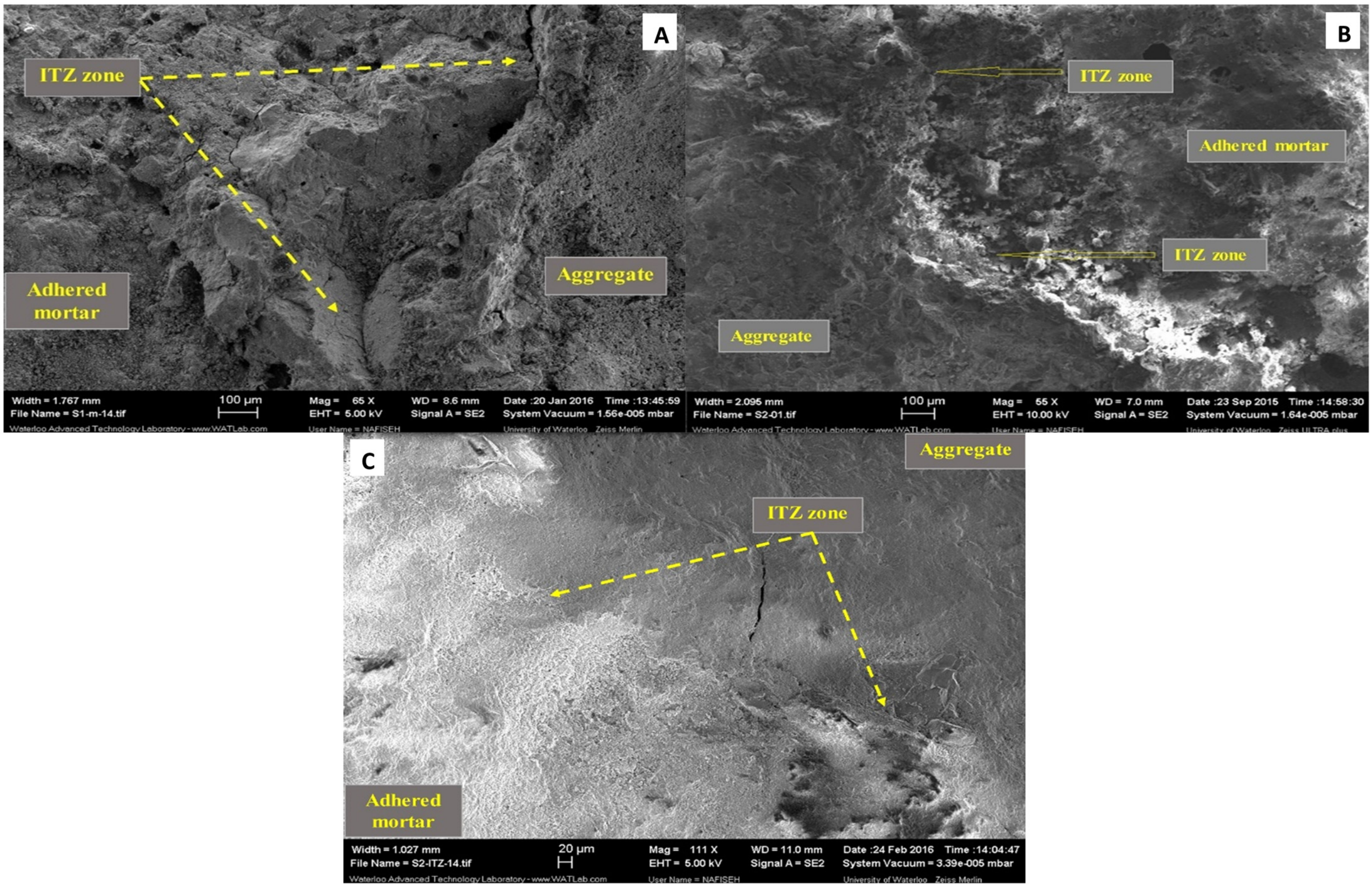

Figure 2. Microstructure of ITZ area for CRCA subjected to heat treatment approach at different temperatures: (**A**) 250 °C, (**B**) 350 °C, and (**C**) 500 °C.

The SEM micrographs of the ITZ between the aggregate surface and the adhered mortar for treated CRCA with acid treatment are presented in Figure 3A,B. According to magnified perspectives, the obtained SEM images showed that acid treatment using weak and strong acids enhances the ITZ. It is interesting to note that the ITZ borderline between the aggregate surface and adhered mortar paste was highly homogenous, uniform, and visible without accumulated CSH particles for CRCA treated with acetic acid. On the contrary, the ITZ borderline of CRCA treated with HCl acid was less homogenous and relatively non-uniform. Directly next to the ITZ borderline, small irregular CSH particles are randomly spread across the adhered mortar surface of CRCA that was treated with acetic acid. On the other hand, the surface morphology of CRCA treated with HCl acid is characterized by a mortar surface that is non-uniform, highly cracked, and porous. Therefore, a considerable difference in the CRCA surface degree of roughness was observed, dependent on the acid treatment type. This method effectively removes adhered mortar, as acidic solutions attack the adhered mortar's surface and dissolve it. It is important to note that the acidic attack had no impact on the aggregate surface for weak and strong acids, indicating that the aggregate type appears unaffected by acidic treatment. It was observed in a previous study that HCl treatment causes significant damage to the CRCA surface compared to acetic acid surface treatment. This can be explained in two ways. Firstly, a substantial difference exists in how strong and weak acids can impact the attached mortar surface. Secondly, the surface material type varies considerably among substances. De Juan & Gutiérrez [37] stated that HCl treatment could not successfully treat RCA-containing limestone aggregates due to the adverse acid attack on this aggregate type.

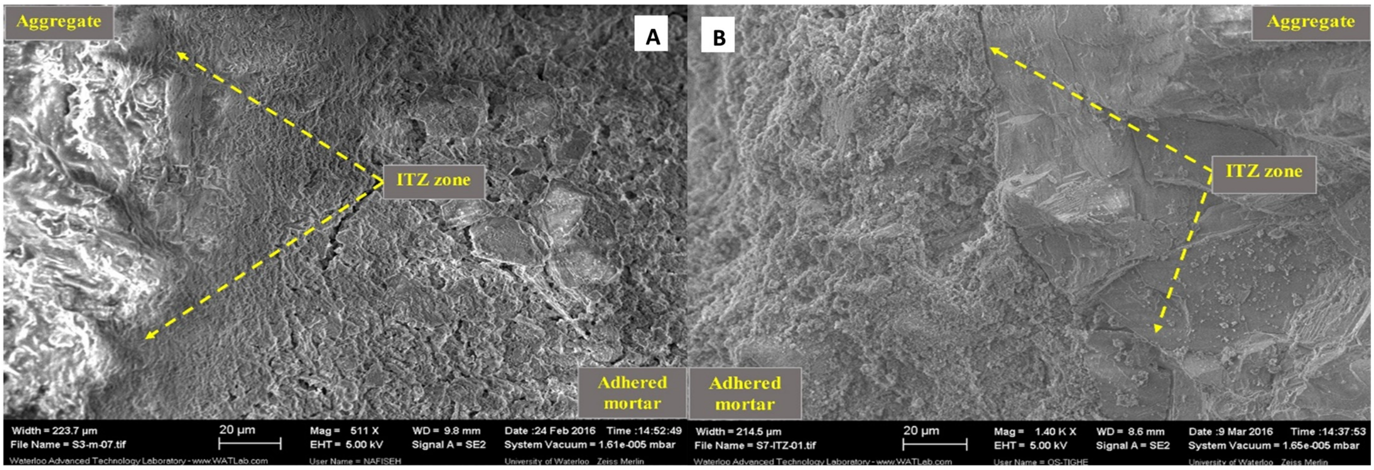

Figure 3. ITZ microstructure of treated CRCA with acid treatment: (**A**) HCl acid and (**B**) acetic acid.

3.2. Chemical Composition for ITZ

For untreated and treated CRCA with various methods, the results of the EDAX analysis concerning the ITZ are shown in Figure S1A–F (Supplementary File). The findings show spectrum analysis for mineral and chemical CRCA composition via EDAX quantification.

3.2.1. Calcium to Silicon (Ca/Si) Atomic Ratio

The EDAX analysis demonstrated that, with rising temperatures during the heat treatment, a significant reduction in the Ca/Si ratio resulted, as shown in Table 1. A significant decrease was seen in the temperature range between 20 °C and 250 °C. The rising temperature removes water molecules from hydrous compounds. This dehydration process leads to increased CSH formation and new dehydrated compounds. The rising temperatures are continuous and have a significant effect on the type of compound that results from the process of dehydration. The thermal gravimetric analysis (TGA) findings provided further details about the relationship between rising temperatures and weight loss due to water removal. At a temperature lower than 105 °C, the vaporized water is related to mass loss and may lead to poorly formed CSH. At temperatures between 105 °C and 200 °C, the water loss results from the dissociation of water. This is linked with factors such as ettringite and lower-temperature CSH, whereas the reason for the loss when temperatures are between 200 °C and 420 °C is because correlated water molecules dissociate into well-formed hydrated compounds such as CAH and CSH [38–40]. Aggregate and RCA type and their chemical composition are acceptable factors influencing the Ca/Si reduction through heat treatment. A high increase in CSH production and significant simultaneous CH consumption are the main reasons for lowering the Ca/Si ratio. The notable CH reduction is due to the new dehydrated composites due to increasing temperatures, especially CSH. This may promote the formation of siliceous and siliceous and aluminous materials, which behave as pozzolans. These pozzolans [21,24] can consume CH accumulated in the pores, producing CSH and decreasing the Ca/Si ratio, as seen in the pozzolanic reaction, as stated previously in Equation (3).

The results obtained from the EDAX analysis also showed that a considerable increase in the Ca/Si ratio is noticeably observed in the temperature range between 250 °C and 500 °C, indicating a significant effect of the heat treatment approach at elevated temperatures on the Ca/Si ratio. Material decomposition and mass loss, including the chemical breakdown in which H_2O and CO_2 are generally obtained from exposure to high temperatures. TGA analyses of RCA revealed a link between weight loss and water separation in well-hydrated CSH at temperatures ranging from 200 °C to 420 °C. $Ca(OH)_2$ breaks down between 420 °C and 550 °C. Moreover, poorly crystalline $CaCO_3$ molecules decompose to release CO_2 at temperatures between 550 and 720 °C, while their highly crystalline molecules disintegrate at temperature intervals of 720–950 °C. Nonetheless, such temperature ranges differ as a result of a number of different characteristics, such as the kind of aggregate and its chemical content.

The values of the Ca/Si ratios of CRCA through acid treatment are also provided in Table 1. It is noteworthy that using the acid treatment method (both acids) resulted in a significant reduction in the Ca/Si ratio in comparison to the thermal treatment approach. This indicates that a highly successful approach is registered for both acid treatment methods compared to the heat treatment technique.

Table 1. Ca/Si atomic ratio of the ITZ area following different treatment methods.

N	Treatment/Property	Ca/Si Ratio
1	Untreated CRCA at 20 °C *	1.95
2	CRCA treated with heat treatment at 250 °C	1.34
3	CRCA treated with heat treatment at 350 °C	1.58
4	CRCA treated with heat treatment at 500 °C	1.75
5	CRCA treated with HCl acid	0.98
6	CRCA treated with $C_2H_4O_2$ acid	0.71

* Normal lab temperature.

3.2.2. Aluminium to Calcium (Al/Ca) Atomic Ratio

The behaviour of the Al/Ca atomic ratio from the outcomes of EDAX analysis through heat treatment is presented in Table 2. It is revealed that a considerable increase is noticed in the atomic ratio of Al/Ca through heat treatment. As the heat treatment does not introduce external sources of Al, the potential reasons for this increase could be due to two things. Firstly, there is a possibility that Al (which can come from raw materials or pozzolanic products) may substitute Si in the CSH compounds. In the model of CSH proposed by Richardson and Groves [41,42], the tobermorite–jennite model, tobermorite-like structural elements are intermixed with jennite-like constituents. This model assumes that Al^{3+} ions only replace Si^{4+} at bridging sites [43–45]. The replacement approach is strongly supported by the obtained EDAX data on the weight percentage of Al, which remains approximately constant or has increased slightly. Secondly, another important issue is that the high amounts of Al may be related to the behaviour of the pozzolanic material. It has been previously reported that different CSH types are formed, such as calcium aluminate hydrate (C_4AH_{13}), with a high amount of Al and a lower Ca/Si ratio if there is a pozzolanic material added, including fly ash from coal combustion, granulated furnace slags, and metakaolin [46]. Then, C_4AH_{13} converts to hydrogarnet (C_3AH_6), as is widely agreed. C_3AH_6 is formed when kaolinite, similar to metakaolin, is used as the source of Al, and the ratios of Al/(Al + Si) are between 0.1 and 0.50 [47]. This approach was used to evaluate the data obtained, as seen in Table 2. In Table 2, the tabulated data suggest that the occurrence of hydrogarnet is highly probable.

It is interesting to note that a considerable difference in the Al/Ca ratio between the strong acid and the weak acid is observed, as can be seen in Table 2. From the point of view of chemistry, it seems reasonable that the reaction between the Cl^- ions and the Al^+ ions in the aqueous solution is more effective than the reaction between acetate (CH_3COO^-) ions and Al^+ ions. The reason is a result of the substantial difference in the corrosive capability of the acids used, strong and weak acids, on the attached mortar surface. This can lead to a large difference in the percentage of the CAH compounds, especially ettringite, within the intermixed phases.

Table 2. Al/Ca and Al/(Al + Si) atomic ratios for the ITZ area of CRCA after various treatments.

N	Treatment/Property	Al/Ca Ratio	Al/(Al + Si) Ratio
1	Untreated CRCA at 20 °C *	0.11	0.17
2	CRCA treated with heat treatment at 250 °C	0.15	0.17
3	CRCA treated with heat treatment at 350 °C	0.29	0.31
4	CRCA treated with heat treatment at 500 °C	0.34	0.38
5	CRCA treated with HCl acid	1.04	0.50
6	CRCA treated with $C_2H_4O_2$ acid	0.21	0.13

* Normal lab temperature.

3.2.3. Intermixed Phases for ITZ

This study used the following criteria to determine intermixed dehydrated phases that are rich in CSH, CH, and monosulphate (AFm) [23,48]:

$$0.8 \leq Ca/Si \leq 2.5,\ (Al + Fe)/Ca \leq 0.2 \tag{4}$$

$$Ca/Si \geq 10,\ (Al + Fe)/Ca \leq 0.4 \tag{5}$$

$$Ca/Si \geq 4,\ (Al + Fe)/Ca > 0.4 \tag{6}$$

where Equations (4)–(6) represent the boundaries of areas rich in CSH, CH, and AFM, respectively.

The atomic ratios of (Al + Fe)/Ca and Ca/Si of CRCA for the heat treatment were calculated based on EDAX outcomes and are plotted in Figure 4. For untreated CRCA (20 °C), the surface of the CRCA is very rich in the CSH phase. It is also indicated that none of the results obtained showed AFm/AFt or CH as the major phases in the ITZ. However, it is still possible for these intermixed phases to exist in reasonable percentages, especially for CH crystals. This is generally thought to be the main reason for the high porosity in the ITZ [23].

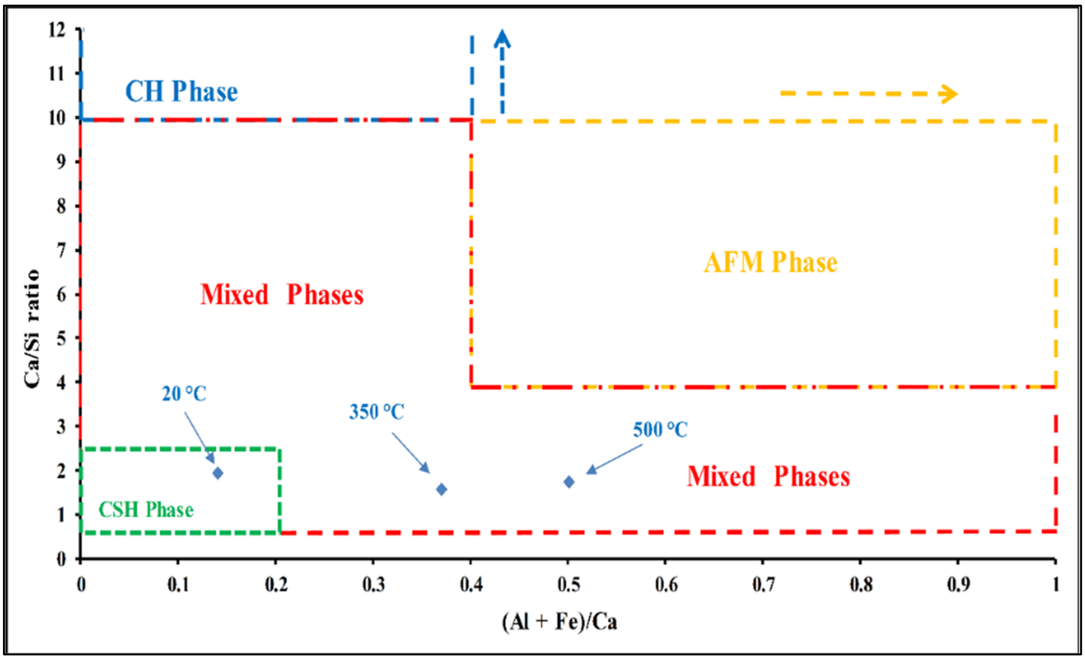

Figure 4. A schematic representation of intermixed phases of ITZ area of CRCA using heat treatment at different temperatures.

It is evident that the (Al + Fe)/Ca and Ca/Si ratios for the heat treatment demonstrate that the intermixed phases undergo a substantial change in the ITZ area. Although the

Ca/Si ratio is decreased, a discernible shift from the CSH phase to the AFM/AFt and CAH phases can be observed. The impact of heat treatment, particularly within the temperature range of 250 °C to 350 °C, can be explained by the likelihood that an elevated proportion of (Al + Fe/Ca) leads to secondary ettringite formation. Ettringite recrystallization was thought to be triggered by releasing a significant quantity of aluminum, iron, and sulphur, as suggested by Erdem et al.'s study [23].

In terms of acid treatment, the EDAX findings revealed that the CRCA surface of the ITZ treated with HCl acid is highly rich in CSH and AFm phases due to the lower Ca/Si ratio and higher sulphur percentage. The surface is comparatively lower in the AFt phase and poor in the CH phase. For the CRCA surface of the ITZ treated with acetic acid, the predominant phases are CSH and AFt, whereas AFm is less dominant.

3.3. XRD Analysis

The XRD analysis findings used to evaluate CSH compounds are presented in Figure S2A–F (Supplementary File).

3.3.1. XRD Analysis for Untreated CRCA

The obtained results indicated that different hydration compounds are found, including tobermorite, jennite, and ettringite, due to the hydration of cement constituents during the lifetime of the concrete. It is important to note that the dolomite percentage was the predominant phase among the different phases, indicating the type of original aggregate. The outcomes of the XRD analysis also showed other compounds, such as calcium carbonate and silicon oxide.

3.3.2. XRD Analysis for Treated CRCA with Heat Treatment

The behaviour of dolomite through heat treatment is presented in Figure 5. A high reduction in the dolomite percentage was observed through the heat treatment. However, a significant decrease was noticeable between 20 °C and 250 °C compared to the range between 250 °C and 500 °C. This refers to a thermal dissociation that leads to different compounds, including CSH transformation. The results also revealed that the behaviour of dolomite strongly correlates with heat treatment and is exhibited as a power law equation due to achieving a high regression value.

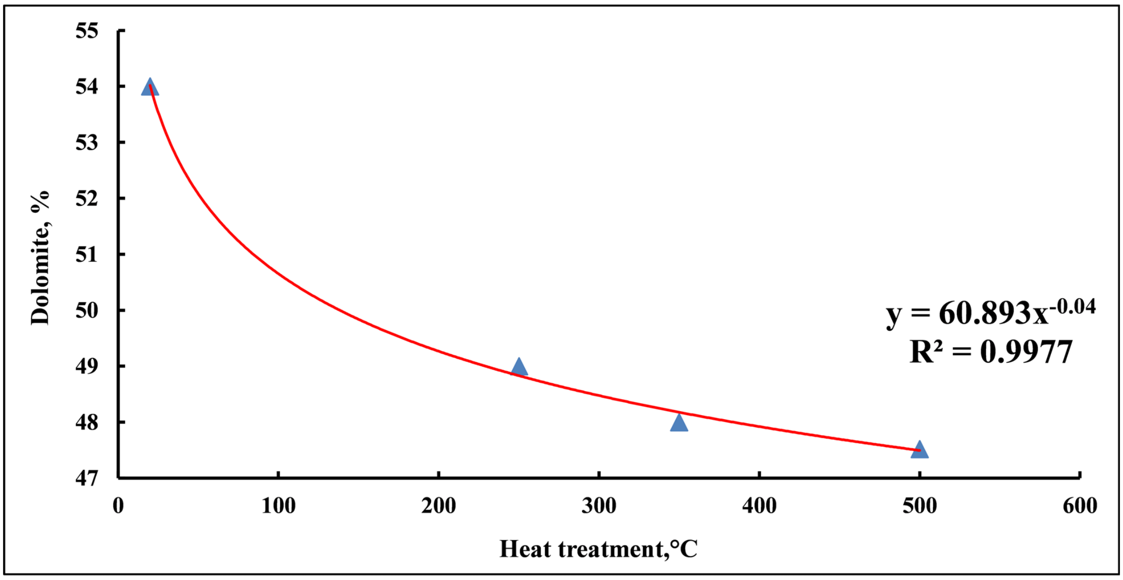

Figure 5. The behaviour of dolomite during heat treatment at different temperatures.

The data obtained from the XRD analysis of the CSH compound, tobermorite, is presented in Table 3. The findings generally revealed a significant increase in the tobermorite percentage for the heat treatment. However, the maximum percentage of tobermorite was registered at 250 °C, whereas a slight decrease compared to the maximum percentage was noticeable, leading to a negative influence of the treatment at elevated temperatures on the tobermorite conversion.

Table 3. Values of CSH compounds: jennite and tobermorite after different treatment methods.

N	CRCA Treatment	Value of CSH Compound, %	
		Jennite	Tobermorite
1	Untreated CRCA at 20 °C *	10.0	11.0
2	CRCA treated with heat treatment at 250 °C	6.0	15.0
3	CRCA treated with heat treatment at 350 °C	7.0	13.0
4	CRCA treated with heat treatment at 500 °C	7.92	13.86
5	CRCA treated with HCl acid	5.88	14.71
6	CRCA treated with $C_2H_4O_2$ acid	14.0	16.0

* Normal lab temperature.

Table 3 also presents the behaviour of the CSH compound, jennite, during the heat treatment. The results demonstrated a significant decrease in the jennite percentage for temperatures ranging from 20 °C to 250 °C. This reduction could be explained by transforming to another type of CSH, namely tobermorite. From the literature, it is known that the CSH jennite type has more water molecules than the CSH tobermorite type. With rising temperatures, the possibility of removing water molecules is highly probable, which can lead to a higher tobermorite percentage. This seems to be quite reasonable due to TGA studies that indicate the removal of water molecules could be found within the temperature range of 20–250 °C, as discussed earlier. On the other hand, this approach can also explain the significant increase in the tobermorite percentage within the same temperature range. Interestingly, a slight increase was observed in the jennite percentage at high temperatures between 250 °C and 500 °C. This increase may refer to the greater conversion and transformation of dolomite to more CSH compounds, including jennite, as it was noticeable in the behaviour of dolomite within the same range of temperatures.

3.3.3. XRD Analysis for Treated CRCA with Acid Treatment

As shown in Table 3, the findings of the XRD analysis indicated many observations that can be mentioned in the following discussion. Though HCl has a strong ability to attack materials, there is a slight dissociation of dolomite to form CSH compounds. It is noticeable that there is a significant reduction in the percentage of the jennite CSH type, indicating a similar behaviour with heat treatment between 20 °C and 250 °C. This large decrease could refer to the sensitivity of the jennite structure to the acidic environment compared to the tobermorite. This appears reasonable due to the significant percentage of tobermorite obtained within the same acidic environment. The XRD analysis of CRCA treated with acetic acid showed a significant conversion of both tobermorite and jennite CSH types compared with the heat treatment and acidic treatment with HCl acid.

3.4. Selection Criteria of Best Treatment Methods

It can be stated that the heat treatment application and both acid treatment methods are highly successful in improving the microstructure of the ITZ. However, using heat treatment at elevated temperatures (500 °C) negatively impacts the ITZ microstructure because of the presence of microcracks on the aggregate surface perpendicular to the ITZ. Additionally, using acidic treatments showed a big difference between HCl and acetic acid for enhancing the microstructure of the ITZ.

The authors' previous studies [6,36] related to enhancing the CRCA's physical properties demonstrated that the heat treatment method is a highly successful technique for improving RCA's various characteristics when the process is performed at temperatures between 300 °C and 350 °C. However, the heat treatment significantly affects the CRCA characteristics at high temperatures.

The authors' investigations also showed that acid treatment at low concentrations efficiently enhances CRCA properties. Nevertheless, acetic acid treatment has a greater effect in improving CRCA characteristics than HCl acid's impact. In addition, acetic acid is safe and preferable due to the highly corrosive effects of HCl acid on some aggregate surfaces. De Juan & Gutiérrez [37] stated that treatment with HCl should not be applied to RCA types that originally consisted of limestone aggregates because of negative acidic attacks on the surface of these aggregate types. Based on this, heat treatment at 300 °C and acetic acid treatment were chosen to be further used with a short mechanical treatment to apply the integration of different treatment techniques.

3.5. CRCA Properties after Dual Treatment

After applying the integration approach between various treatment methods, the outcomes of the significant properties of CRCA are presented in Table 4. The findings indicate two different stages of CRCA treatment. In the first stage, the CRCA treatment involved the application of two different approaches: pre-soaking in the weak acid solution and heat treatment. The second stage included utilizing short mechanical treatment using the Micro-Deval device. As different treatment techniques were used, the comparison with the untreated CRCA appears reasonable to obtain a fair evaluation of the treatment methods.

Table 4. Main CRCA properties after different dual-treatment approaches.

Characteristic	Untreated CRCA	Dual-Treated CRCA Approach I *	Dual-Treated CRCA Approach II **
Bulk Relative Density (BRD)	2.421	2.486	2.551
Absorption, %	3.74	2.879	2.36
Porosity, %	9.05	7.16	6.02

* This approach includes the integration of treatments: heat and short-term mechanical treatment. ** This approach involves the integration of treatments: soaking in weak acid solution and short-term mechanical treatment.

As shown in Table 4, a significant improvement in CRCA is recorded due to the impact of treatment methods. However, different treatment approaches seem to affect CRCA characteristics differently. Considerable improvement is obtained for BRD and water absorption properties under the influence of treatment techniques. Because of the integration of short mechanical treatment and heat treatment at 300 °C, the water absorption of CRCA was lowered from 3.74% to 2.88%. This treatment approach leads to a reduction of 23.0% in this vital characteristic. Meanwhile, CRCA water absorption is considerably reduced from 3.74% to 2.36% due to the integration of two treatments: the treatment with an acetic acid solution and the mechanical method. Due to the application of this approach, a considerable reduction of 37% is registered for water absorption, indicating the best performance between different treatment methods.

Simultaneously, there is a slight improvement in the BRD property for different integration types of treatment methods. However, it is essential to mention that using the pre-soaking method and short mechanical treatment seems more efficient in enhancing this property. From the perspective of the porosity property, the experimental findings demonstrated a considerable improvement in this property due to applying different treatment techniques. There is a significant reduction, approximately 20.9%, in the porosity of CRCA after using the integration of heat treatment at a temperature of 300 °C followed by the mechanical treatment technique. The porosity value was significantly reduced when the pre-soaking procedure was used with the same mechanical treatment. Thus, this treatment approach is highly effective for improving the porosity of CRCA, with an estimated decrease of 33.5%.

To achieve a preferable understanding, the laboratory results of water absorption and BRD are further evaluated by comparing them with RCA literature studies. The values of the characteristics mentioned above are graphically presented in Figure 6. From the mentioned figure, it can be stated that most RCA studies refer to the extent of absorption ranging between 4% and 8%. However, extreme values can be found outside of that extent, with water absorption values ranging between 9 and 10%. In addition, the majority of RCA research indicates that RCA's specific gravity typically falls between 2.2 and 2.6. Nevertheless, there are instances when the specific gravity levels of RCA might exceed the mentioned range, reaching as high as 2.7–2.9, as presented in Figure 6. Based on this, the obtained values of BRD and water absorption of untreated CRCA are classified within the first half of the extent mentioned above. This refers to a suitable CRCA type used in the present study compared to RCA literature studies. It is noteworthy that the integration strategy, which incorporates various treatments, is particularly beneficial in improving the unfavourable attributes of CRCA. Such a strategy is an intriguing fact to take into consideration. According to the results, the main properties of CRCA were significantly improved using a dual-treatment approach. Combining different treatments enhanced the characteristics of treated CRCA, including water absorption and specific gravity, far more than those of untreated CRCA, as shown in Figure 6. However, combining soaking with the acetic acid solution and mechanical treatment is more effective than other integration techniques.

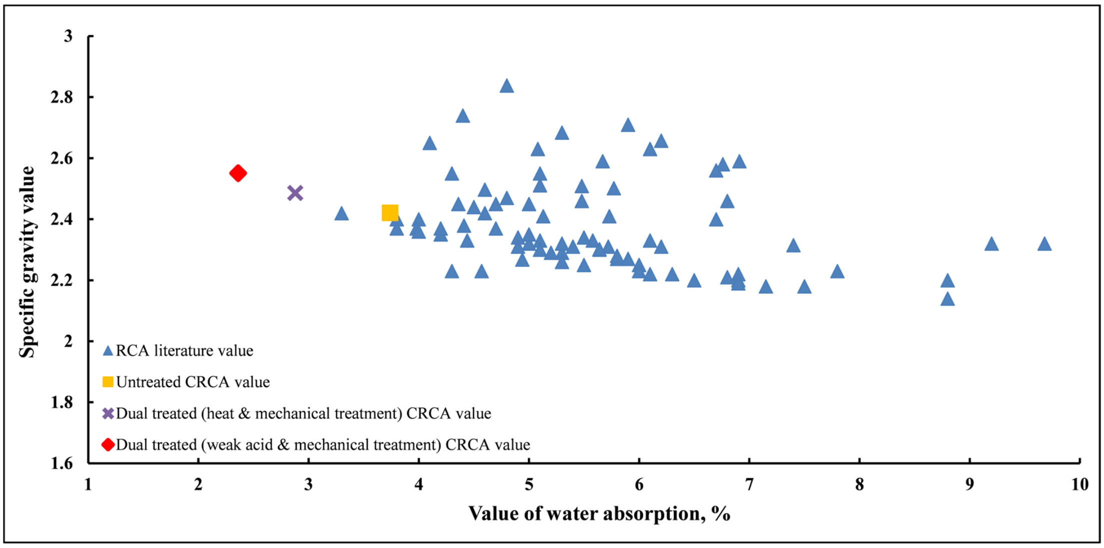

Figure 6. Main properties of dual-treated CRCA: water absorption and specific gravity were obtained in this study compared with RCAs literature studies.

3.6. Performance of CRCA-Containing Asphalt Mixtures

3.6.1. Effect of CRCA Addition on Thermal Cracking

Table 5 presents TSRST results for various asphalt mixtures, which encompass varying proportions of untreated and treated CRCA with various treatment procedures. The obtained results are explained in detail in the following sections.

Table 5. Results of the TSRST of different asphalt mixtures that included CRCA.

Mixture Property	Control Mix, 0% CRCA	Untreated 30% CRCA	Dual-Treated 30% CRCA Approach I *	Dual-Treated 30% CRCA Approach II **	Untreated 60% CRCA
Fracture temperature, °C	−31.5 −31.1 −28.1	−25.95 −24.43 −25.86	−27.37 −26.93 −27.19	−26.77 −26.19 −26.30	−28.16 −24.65 −25.40
Average value	−30.2	−25.4	−27.16	−26.42	−26.07

* This approach includes the integration of treatments: heat and short-term mechanical treatment. ** This approach involves the integration of treatments: soaking in weak acid solution and short-term mechanical treatment.

Influence of CRCA Proportion on the Fracture Temperature

As seen in Table 5, it was revealed that the CRCA addition to the asphalt mixture with various proportions influences the mixture's fractural stress and temperature. Figure 7 shows the average fracture temperature values of the asphalt mixtures with various proportions of untreated CRCA addition. The addition of untreated CRCA to asphalt mixtures increases the average fracture temperature compared to the control mixture without CRCA. This is due to the fact that RCA has more micro-cracks than Nag. The presence of microcracks leads to RCA particles having lower strength than Nag particles. In addition, the adhered mortar particles could be more brittle under continuous low temperatures, resulting in a high possibility of more microcracks appearing and, consequently, rapid failures [47]. It is demonstrated that the highest increase in the fracture temperature was 2.6 °C, which was higher than the comparable low-temperature performance grade of the asphalt binder utilized, PG 64-28 (−28 °C).

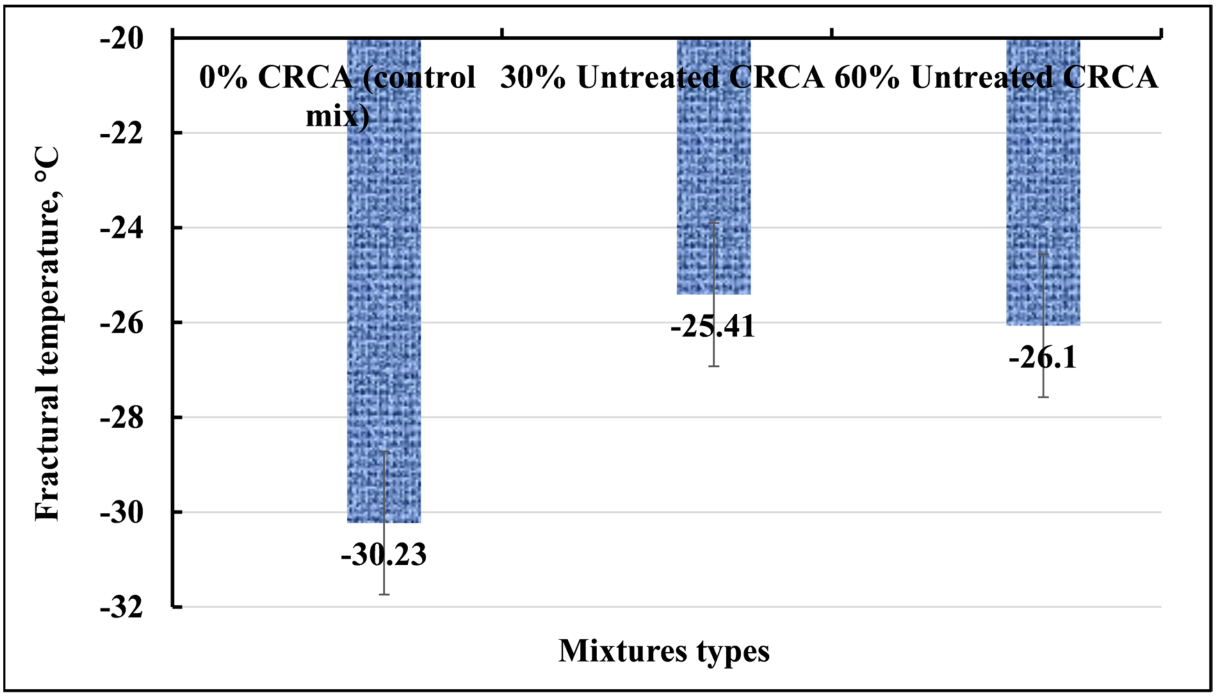

Figure 7. Fracture temperature of mixtures containing various proportions of untreated CRCA.

Meanwhile, it is interesting to note that an increased CRCA proportion of up to 60% led to only a small reduction in the fracture temperatures of HMA mixtures. Such behaviour is attributed to the rough surface of CRCA, which has several crushed faces that become more prevalent as the quantity of CRCA added rises. Such surfaces lead to an increased contact area between the CRCA and the asphalt binder. Thus, more abrasion force is generated [49], providing greater interconnecting forces that resist the impact of low temperatures on the asphalt mixture. Based on the obtained results, it can be stated that the asphalt mixtures that include CRCA have good behaviour related to the resistance of low-temperature cracks, resulting in a successful application in low-temperature regions. Nevertheless, it is essential to mention that the mixes above appear unsuitable for areas with severe weather conditions. Examples of these areas are the Northern Canadian regions, where temperatures are extremely low and could reach between −30 °C and −40 °C with long winter periods.

Effect of Treated CRCA on the Fracture Temperature

The average fracture temperatures of different mixtures that included treated CRCA with various treatment approaches were used to evaluate the influence of the integration of different treatment types on thermal cracks, as presented in Figure 8. Interestingly, the integration of short-term mechanical treatment and heat treatment at 300 °C significantly reduced the fracture temperature. The average fracture temperatures for mixtures containing 30% of treated and untreated CRCA were −25.41 °C and −27.2 °C, respectively, exhibiting a reduction of 6.7% in fracture temperature. The obtained outcome (−27.2 °C) was higher by only 0.8 °C than the comparable low-temperature performance grade of the associated asphalt binder (−28.0 °C). As a result, this treatment method appears to be quite effective and practical in low-temperature regions. Such an outcome could be due to the improved treated CRCA, as shown earlier based on its physical properties. Additionally, treated CRCA registered a more significant improvement in terms of the ITZ's surface morphology and texture between the adhered mortar and aggregate surface compared to untreated CRCA. As mentioned previously, such improvement was observed for treated CRCA at temperatures of 250 °C and 350 °C due to accumulated dense particles in the ITZ area.

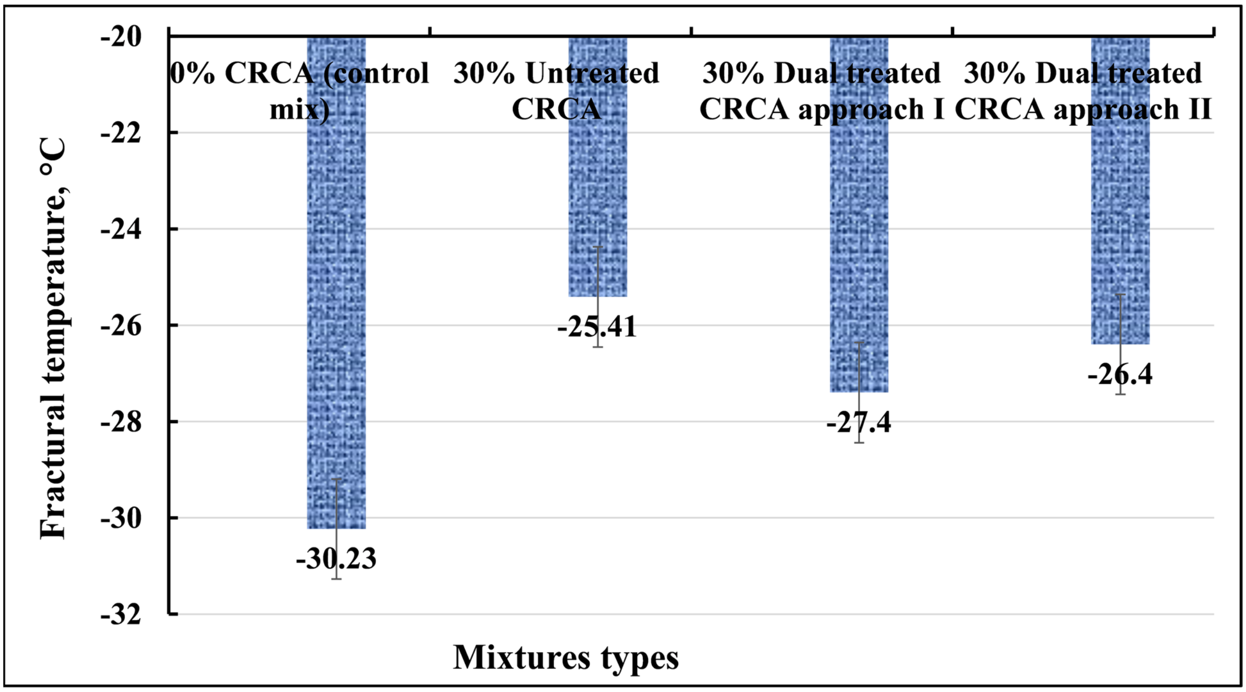

Figure 8. Effect of treated CRCA with different approaches on the fracture temperature of asphalt mixtures.

In terms of the integration of pre-soaking in acetic acid and short-term mechanical treatment, the average fracture temperature for the mixture that included 30% treated CRCA was −26.4 °C compared to the mix with 30% untreated CRCA, which had a fracture temperature value of −25.4 °C. This outcome was higher than the corresponding low-temperature performance grade (−28.0 °C) of the respective asphalt binder by 1.6 °C. Consequently, this integration approach appears to be successful for cold regions. However, the obtained results were still higher than the fracture temperature of the control mix.

3.6.2. Evaluation of Tensile Strength of Asphalt Mix

The ITS test is usually used to measure the tensile strength of asphalt mixtures. It could be further used to evaluate relevant behaviours such as road surface cracking, permanent deformation, and stripping [49]. Figure 9A illustrates the conduct of the ITS values of asphalt mixes that include different specimens: conditioned and unconditioned. The findings revealed that the ITS values of samples are generally greater than those of the 0% untreated CRCA mix (control mix). This led to favourable outcomes for different percentages of untreated CRCA, including a substantial proportion of 60% CRCA. It is noteworthy that the highest ITS values documented for mixtures containing 30% untreated CRCA are 941.5 kPa for unconditioned samples and 856.5 kPa for conditioned mixes. These values represent an increment of 85.6% and 86.7%, respectively, compared to control mix values. Subsequently, the ITS values of the mixture containing 60% untreated CRCA increased by about 72% and 57% for unconditioned and conditioned specimens, respectively. This behaviour is confirmed by previous studies [50]. Other experiments, on the other hand, have indicated that the value of ITS rises when the fraction of RCA increases [49].

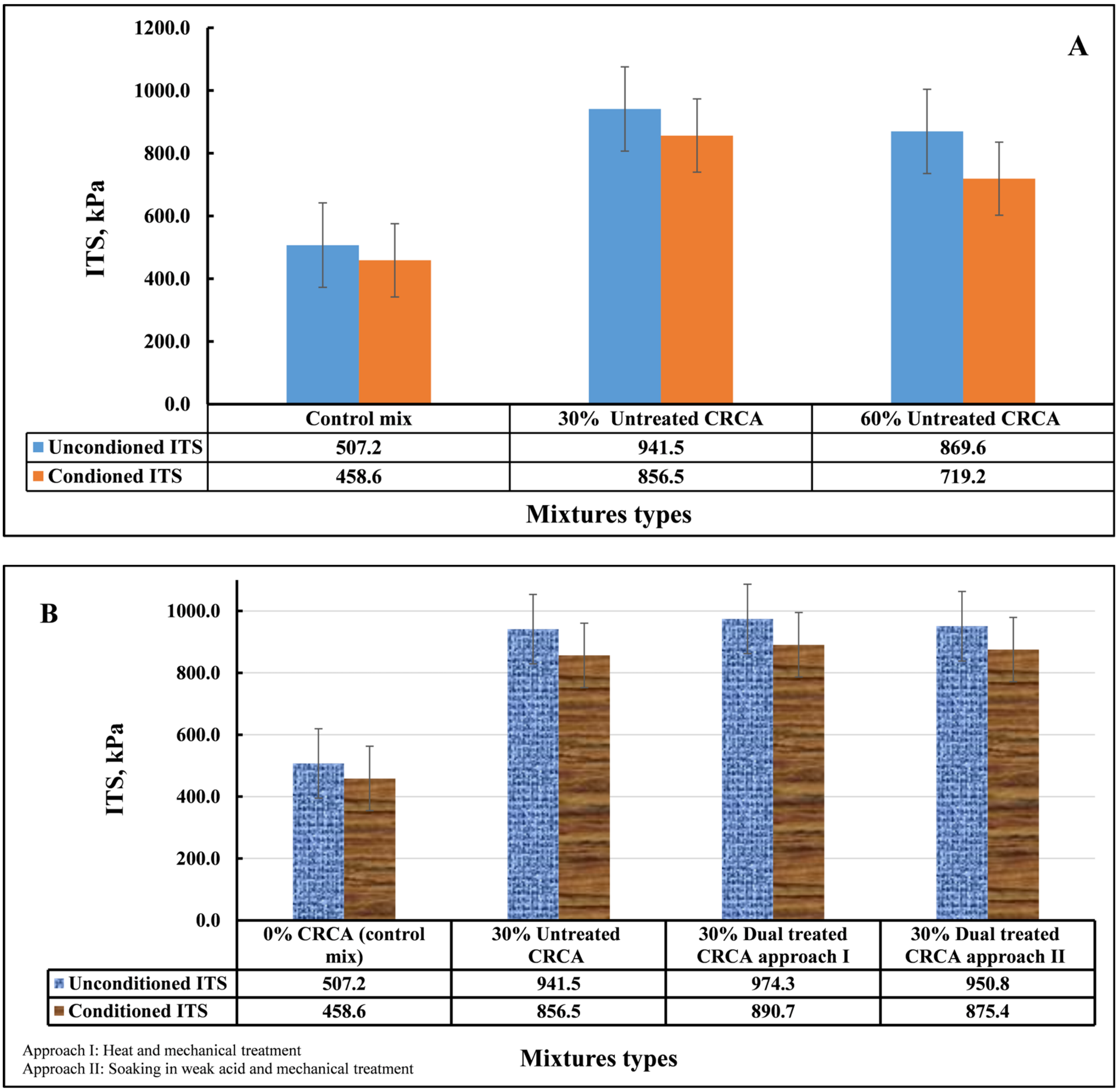

Figure 9. ITS values for asphalt mixtures containing (**A**) varying fractions of untreated CRCA and (**B**) treated CRCA using various treatment methodologies.

Figure 9B illustrates the assessment of the impact that different treatment approaches have on ITS values. The results of the experiments showed that the ITS values recorded for mixes that included 30% treated CRCA with various treatments were significantly higher than those recorded for the mixture that contained 30% untreated CRCA. Due to the integration of heat and short-term mechanical treatment, the ITS values of both unconditioned and conditioned registered an increase of 3.5% and 4.0, respectively. This indicates that this integration type has the best performance for improving ITS values compared to other combination treatment approaches. Though the integration of soaking in weak acid and a short-term mechanical treatment showed negligible improvement in the ITS value of the unconditioned mixture, with an increase of 1.0%, this type of treatment integration provided little improvement for the conditioned mixtures, with an increase of only 2.2%.

3.6.3. Evaluation of Moisture Sensitivity

Moisture sensitivity, referred to as moisture damage, pertains to a form of deterioration wherein the mechanical properties of an asphalt mix are predominantly impacted by the existence of water [51]. In asphalt mixtures, moisture damage is known as the loss of stiffness and strength due to moisture exposure under mechanical loading, and its appearance is generally due to a phenomenon named stripping. Moisture damage significantly impairs the performance of pavement, which is a critical factor contributing to various forms of distress, including rutting and fatigue cracking. To achieve an HMA capable of effectively resisting damage caused by moisture and water, the minimum value of necessary TSR should fall between 70% and 80% [10,52]. Prior studies carried out using RCA suggested that it would be beneficial to provide the asphalt mix with an adequate amount of time at elevated temperatures to ensure that the binder is fully absorbed by the aggregates. This is because the bitumen absorbed by the RCA effectively covers the whole aggregate surface, preventing water from seeping through cracks or openings. Therefore, this could be a technique to increase the moisture sensitivity of the asphalt mix that included RCA. In addition, it decreases the porosity while simultaneously reducing the number of spaces that are accessible to water [53–55].

Figure 10A illustrates the variation in TSR values for mixes containing varying proportions of untreated CRCA. According to the findings, all of the TSR values were greater than 80%, the minimum value needed by the MTO standard for asphalt mixes. These outcomes demonstrated that the mixtures containing varying proportions of untreated CRCA exhibited an exceptional degree of success. The values of TSR decreased with the increase in CRCA addition. It is interesting to note that the mixture containing 60% CRCA had the lowest TSR value among all the mixtures tested. This could be attributed to poor adhesion of CRCA with asphalt when the CRCA proportion is significantly increased [50]. Additionally, the outcomes revealed that a mixture containing 30% CRCA exhibited a marginally greater moisture sensitivity than those containing just Nag. In light of the moisture sensitivity test being particularly significant for nations that experience cold weather, the obtained findings are quite encouraging and meaningful for RCA usage. The obtained results confirmed previous investigations [10,51,56]. In addition, the experimental findings showed that all TSR values for mixes, including different kinds of untreated CRCA with varying percentages, are higher than the minimum criteria for a previous study, registering an efficient outcome for the mixes that included RCA [57]. In contrast, an opposite behaviour for TSR values was found in other studies [49,52,53]. This contradiction could refer to other factors, including RCA type and its characteristics; hence, more investigation is required.

Figure 10B shows the influence of treated CRCA with different treatment approaches on TSR values. Compared to the mixture with 30% untreated CRCA, the results showed that a slight improvement in the TSR values is registered for mixtures that included 30% treated CRCA with various treatment methods. However, the obtained findings were much higher than the minimum required TSR value for MTO specifications. It was noted that the integration of the heat treatment method and short-term mechanical treatment has a negligible improvement, 0.44% in the TSR value of the mixture that included 30% treated CRCA. Due to the treatment combination of soaking in weak acid and the same mechanical treatment, the TSR value was slightly increased by only 1.2%. Hence, this integration type has the best performance in improving the TSR value compared to another combination approach. Thus, it is possible to assert that the results of the moisture sensitivity test appear to be in contrast to the results of the ITS test with respect to the effect of the kind of dual treatment integration used.

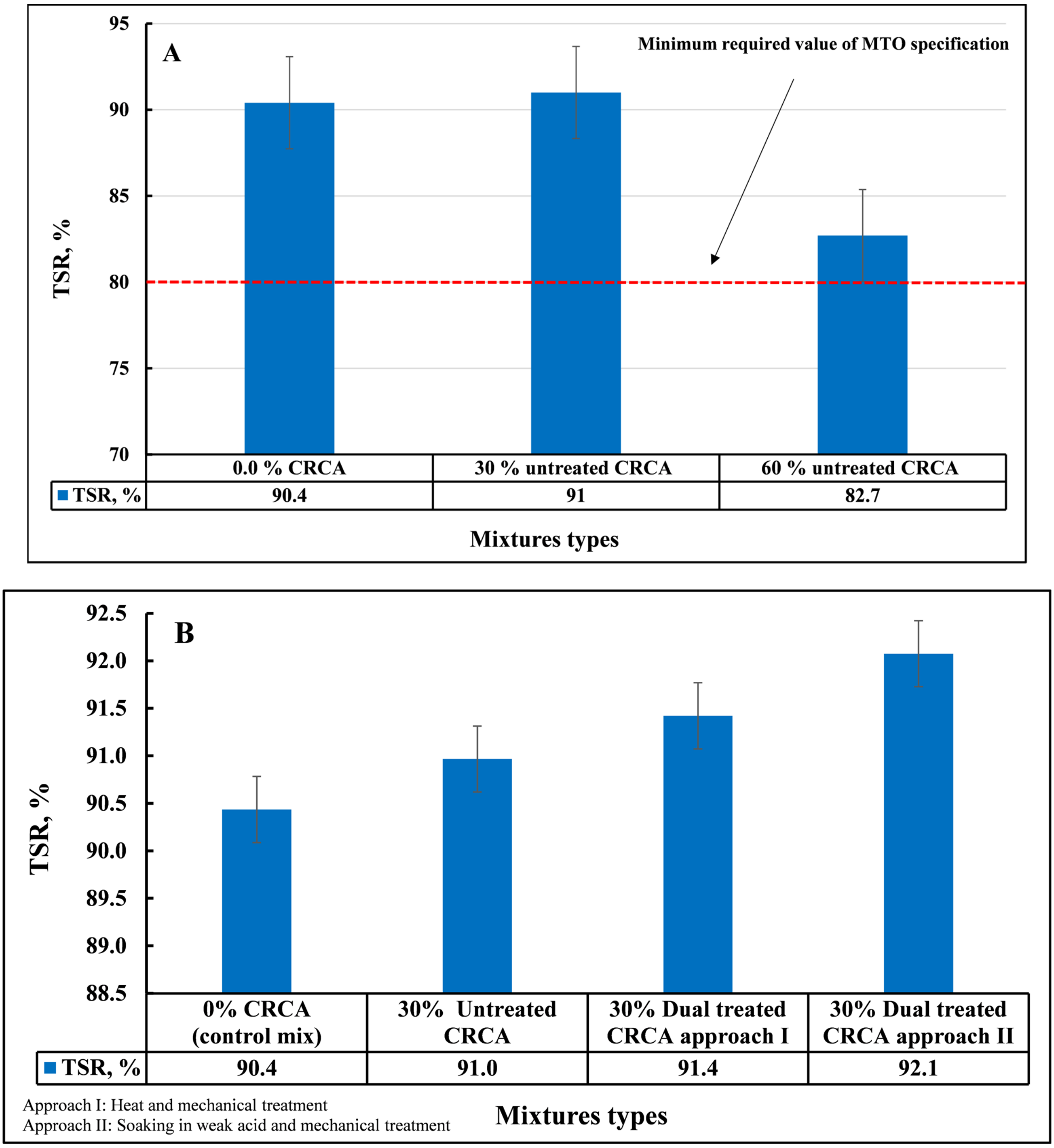

Figure 10. TSR values for mixtures that included (**A**) untreated CRCA at different proportions and (**B**) CRCA treated with different treatment approaches.

3.6.4. Evaluation of Permanent Deformation

In asphalt pavement, permanent deformation, also known as rutting, generally indicates permanent depressions on the asphalt surface that can occur in the paths of pavement wheels [58]. Permanent deformation could exist in the HMA or underlying asphalt layers [59]. Three main reasons can lead to rutting in asphalt pavements: permanent deformation in the subgrade layer, permanent deformation accumulated on the surface layer, and surface wearing pavements due to studded tyres. However, permanent deformation could occur under the influence of all the causes mentioned above [60].

The findings of the wheel track test are shown to be representative of the link between the number of cycles and the accumulative HMA deformation with respect to rutting depth, as displayed in Figure 11A. As generally expected, the permanent deformation of asphalt mixtures increases with increased cycles [51]. According to the majority of studies, asphalt mixes that include RCA are considered to be in compliance with the requirements of permanent deformation [52–54,61]. From Figure 11A, it is crucial to acknowledge that the control mixture, which contains 0% CRCA, exhibits the most severe manifestation of permanent deformation among the rutting levels of different mixtures. Such an outcome demonstrates that the adhesion between the asphalt binder and the CRCA particles is far higher than the adhesion that exists between the similar binder and Nag particles. Such conduct may be accounted for by the uneven surface of CRCA, which exhibits numerous pulverized faces. These surface characteristics provide an extended contact area between the asphalt binder and the CRCA particles, which in turn creates a greater abrasion force. This force may produce a significant interconnect force, which is necessary for the asphalt mixture to be able to withstand the impact of wheel load [49]. As a result, the asphalt mixtures with different CRCA proportions seem to have better permanent deformation conduct than the control mix (0% CRCA), even with a high CRCA percentage of 60%. Such behaviour was also observed in other studies [62–64].

In addition, the findings demonstrated a marginal variation in the resistance to permanent deformation even though the content of CRCA in the mixes rose from 30% to 60%. This resulted in little impact of the CRCA proportion on the permanent deformation of the asphalt mixture. However, there are two possible reasons behind this. It is generally known that the asphalt content of a mixture increases as the CRCA percentage is increased. Firstly, a high asphalt content can provide greater plastic flow susceptibility. Due to high asphalt content, the high plastic susceptibility can lead to losing the internal friction among aggregate particles. Hence, the wheel loads are carried and resisted by the asphalt content only instead of the structure aggregate particles, increasing the permanent deformation of the asphalt mixture [62]. Secondly, a high proportion of CRCA leads to a significant loss of adhered mortar due to the compaction of wheel loads. This results in poor adhesion between the asphalt binder and the CRCA particles. Nevertheless, untreated CRCA in varying proportions exhibited effective incorporation into asphalt mixes compared to only Nag mixes, owing to a substantial enhancement in resistance to permanent deformation. This result confirms previous investigations [49,65–67]. However, according to other studies, as the amount of RCA in the mix rises, the asphalt mix's performance in terms of permanent deformation deteriorates [52]. Some argue that the mix gradation also plays a role in permanent deformation behaviour [68].

To achieve a better understanding, the permanent deformation results are further evaluated in Figure 11B. As can be seen, Figure 11B reveals the permanent deformation of different mixtures that included 30% untreated CRCA and CRCA treated with various treatment methods. A reduction in permanent deformation was highly noticeable for the 30% CRCA mixture treated with heat treatment followed by a short mechanical treatment (approach I), compared to the mix that included untreated CRCA at 30%. This demonstrated a highly successful integration approach of different treatments for enhancing the mixture's resistance to permanent deformation. In contrast, the mixture that included 30% treated CRCA with soaking in acetic acid and short-term mechanical treatment (approach II) had greater permanent deformation than the mix with 30% untreated CRCA. This integration technique seems less successful than the previous combination approach. However, the permanent deformation of the mix that included treated CRCA under the influence of this technique is still much less than that of the mixture that contained 0% CRCA (control mix). Regardless of treatment type, asphalt mixtures with CRCA treated using different treatment approaches appear to have good permanent deformation behaviour depending on the treatment type compared to the control mix (0% CRCA). Such outcomes are highly encouraging and can contribute to increased RCA usage in asphalt mix applications without hesitation.

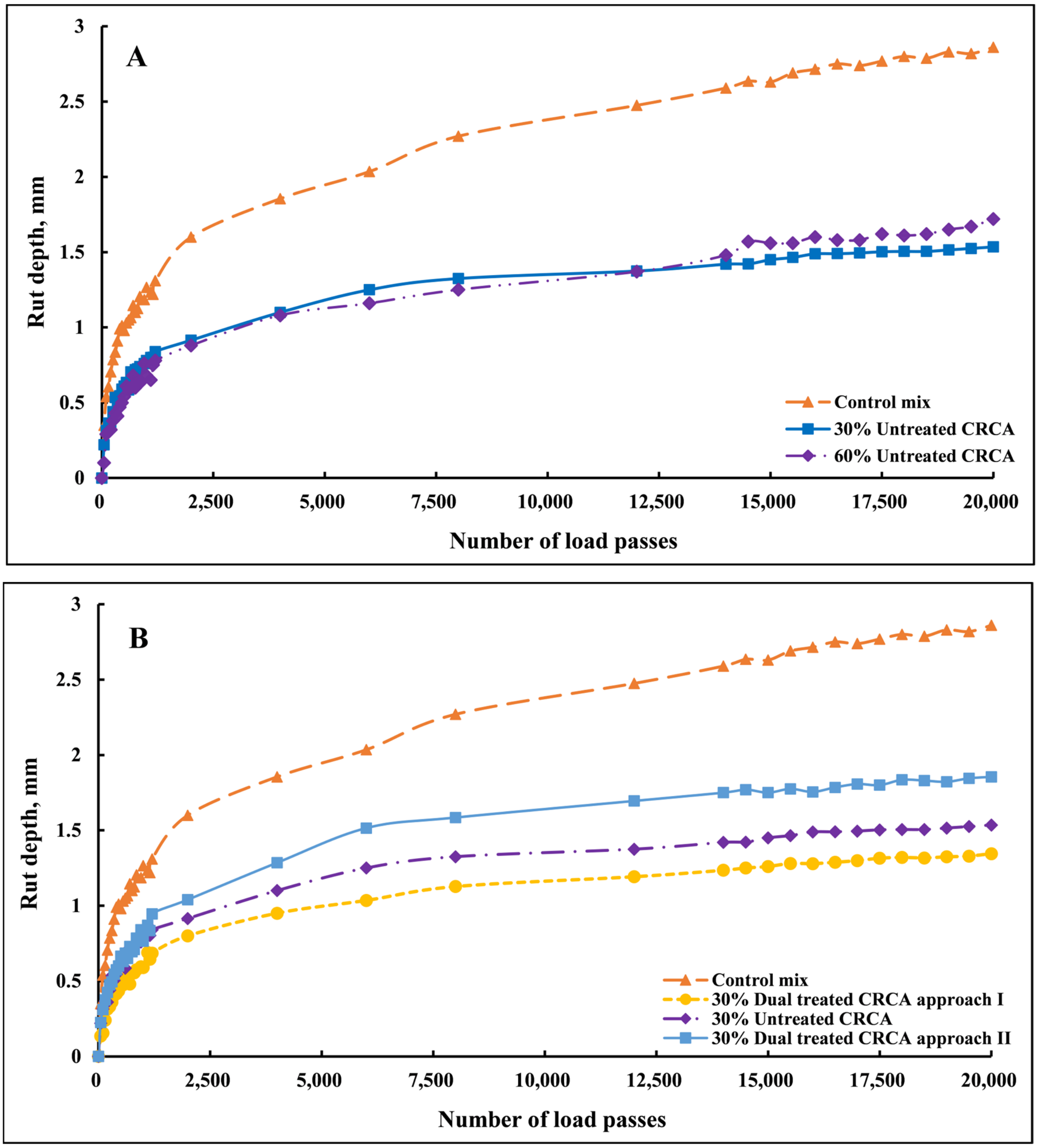

Figure 11. HWRT rut depth of mixtures that included (**A**) various proportions of untreated CRCA and (**B**) CRCA treated with different approaches.

4. Materials and Experimental Methods

4.1. Materials

Natural aggregate (Nag) and one kind of filler, known as dust plant, were provided by the Miller Group. On the other hand, the RCA substance used in this research was classified as granular A, produced by the Steed and Evans company in St. Jacobs, Ontario. From the related literature, it is commonly accepted that CRCA has less residual mortar than fine recycled concrete aggregate (FRCA) [37,69]. Based on this, CRCA seems to have higher quality characteristics; hence, its application is more likely to be successful; than FRCA. From the perspective of economy and success possibility, improving the characteristics of a comparatively good quality material compared to a poor-quality material is a quite reasonable approach. Due to this reason, the current study mainly deals with the CRCA portion of RCA. The CRCA in this study denotes the proportion of RCA that remains on the sieves within the range of 4.75 to 19 mm. The sieve analysis of Nag and RCA is provided in Figure 12. The results of various tests and procedures conducted on untreated CRCA and Nag are shown in Table 6, presenting the mechanical and physical characteristics of these materials.

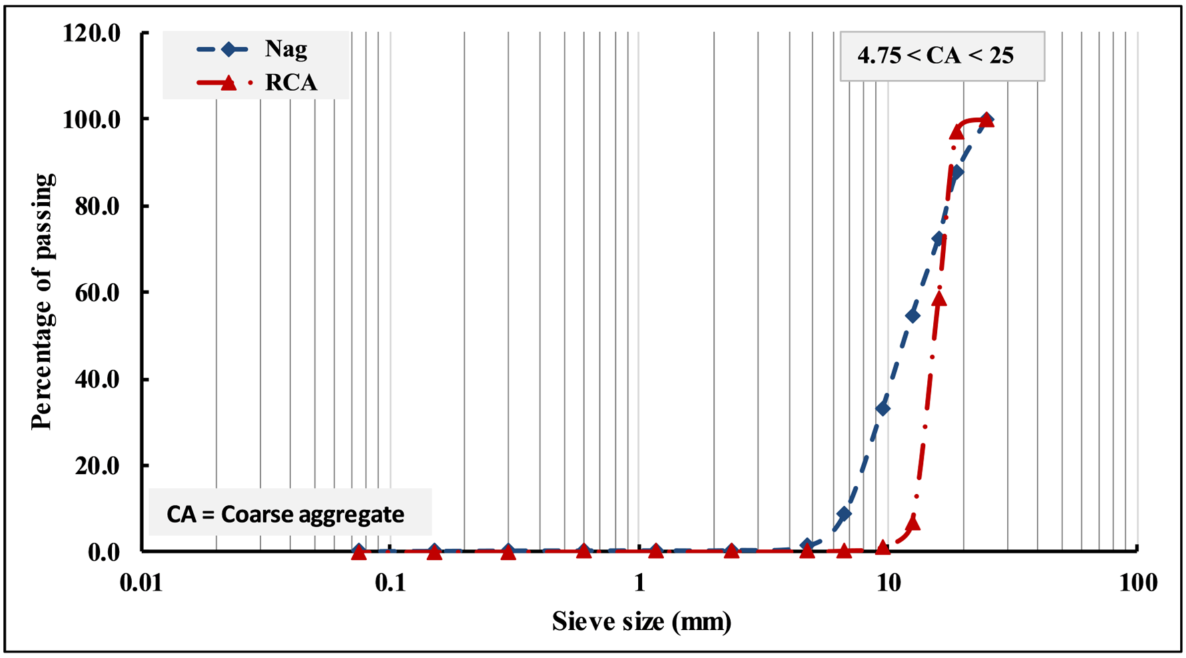

Figure 12. Particle size gradations of Nag and RCA.

Table 6. Properties of Nag and untreated CRCA.

N	Property Category	Property Name	Value		Specifications
			Nag	CRCA	
1	Physical properties	Bulk specific gravity *	2.658	2.421	ASTM C127
2		Water absorption (%)	0.773	3.740	
3	Mechanical properties	Abrasion resistance (%)	15.893	16.031	ASTM D6928
4		Fractured particles (%)	95.501	95.720	ASTM D5821
5		Aggregate crushing value (%)	19.49	23.28	BS 812-110

* The value of this property is not represented as a percentage.

In order to establish the design of the included CRCA mixes, various percentages were used for the incorporation of CRCA into mixtures, which were 0%, 30%, and 60%. To satisfy the mix design requirements of both the Miller Group and the Ministry of Transportation Ontario (MTO), the mixture's graduation was adjusted depending on the percentage of added CRCA. The gradation of Nag and different proportions of CRCA, Miller Group requirements, and MTO specifications are presented in Table 7. The CRCA was subjected to a rigorous washing process to eliminate any surface impurities that had adhered. Prior to conducting different kinds of treatments and tests, the CRCA particles underwent a drying process in a regular oven at a temperature of 105 ± 5 °C for a duration of 24 h. Although the testing of untreated CRCA was carried out at a room temperature of 20 °C, several treatment techniques and investigations were carried out in a variety of settings.

Table 7. Gradations of mixtures with different CRCA proportions, Miller Group requirements, and MTO specifications.

Sieve Size, mm	CRCA's Percentage of Passing, %			Mix Design Target *	Limitations of MTO
	0.0%	30%	60%		
25	100	100	100.00	100	100
19	95.2	96.6	97.32	96.8	90–100
16	89.0	86.6	82.92	90.6	23–90
12.5	81.8	73.9	64.06	83	
9.5	73.2	67.8	59.12	73.3	
6.7	63.3	61.5	55.12	63.3	
4.75	57.1	56.0	51.69	55.9	
2.36	42.8	41.8	40.96	43.5	23–49
1.18	30.7	31.2	30.53	32.5	
0.6	22.9	24.3	23.59	25.1	
0.3	10.2	10.3	10.30	11.8	
0.15	5.4	5.5	5.54	5.5	
0.075	2.1	2.1	2.13	3.8	2–8

* Miller Group requirements.

4.2. Experimental Methods

The implementation of several different treatments in various conditions was carried out to evaluate each treatment separately, followed by an analysis of the integration of numerous treatment techniques. The impact of various treatment methods on the enhancement of ITZ was also investigated. The ITZ enhancement was evaluated by investigating many features, including surface morphology, chemical composition, and intermix phases, for CRCA under the influence of different treatment types. To successfully achieve this, highly advanced techniques and tools, namely SEM, an energy-dispersive X-ray analyzer (EDAX), and X-ray diffraction analysis (XRD), were utilized. In addition, the asphalt mixes were formulated with a single kind of binder, PG 64-28, and a nominal maximum aggregate size (NMAS) in accordance with Superpave regulation requirements. The experimental methods and their related details are discussed as follows.

4.2.1. CRCA Treatment Approaches

In this research, various approaches were applied for treating CRCA, including pre-soaking with different acids, heat treatment at different temperatures, and mechanical treatment. Regarding the pre-soaking approach, two different acids were used: acetic acid ($C_2H_4O_2$) and hydrochloric acid (HCl), considered weak and strong acids, respectively, provided by Sigma-Aldrich. The CRCA samples were soaked in acidic solutions for 24 h at room temperature (20 °C). A low concentration of acidic solution (0.1 M) was used to obtain appropriate acidity settings. After the CRCA particles were subjected to acidic treatment, the specimens were soaked in distilled water and eventually drained to eliminate the residual impacts of the acidic solution. The treated samples were subsequently dehydrated at a regular oven temperature of 105 ± 5 °C to set them up for further examinations. The heat treatment procedure involves subjecting the CRCA specimens to temperatures varying from 250 °C to 500 °C in a conventional oven for a duration of 1 h. To gain a dual-treatment approach, the CRCA specimens that had been heated and pre-soaked underwent further mechanical treatment. The treated CRCA specimens were positioned in a Micro-Deval apparatus in dry conditions for 15 min after applying steel balls. Then, the dual-treated CRCA samples were washed gently with water at 20 °C. The dual-treated samples were then dehydrated in a regular furnace set at 105 ± 5 °C for 24 h. Subsequently, the CRCA samples were filtered through a 4.75 mm sieve to obtain the required coarse portion.

4.2.2. Examination of ITZ

SEM Analysis

The surface morphology and microstructure of ITZs for untreated and treated CRCA with various treatment types and under different conditions were examined using SEM analysis (Zeiss Ultra Plus microscope) supplied by (Carl Zeiss Canada Ltd., Toronto, ON, Canada).

Elemental Analysis for ITZ

To scan the ITZs for CRCA samples, the SEM was provided with an EDAX spectrometer (Carl Zeiss Canada Ltd., Toronto, ON, Canada) to evaluate the elemental composition of the ITZs for untreated and treated CRCA specimens. To perform SEM and EDAX tests, an approximate size of 10 mm of specimens were prepared. The CRCA samples were dried and then coated with a thin layer of gold prior to their examination. SEM and EDAX analyses were conducted in the Waterloo Advanced Technology Laboratory at the University of Waterloo, Canada.

XRD Analysis

XRD analyses were performed using a Panalytical Empyrean diffractometer (Malvern Panalytical, Saint-Laurent, QC, Canada) equipped with a Cu-X-ray tube and a PIXcel 3D detector, operated at 45 kV and 40 mA. Each sample was loaded into two XRD sample holders (with a diameter of 27 mm) using the back-loading technique. Data were collected in Bragg–Brentano geometry with a continuous scan over the 2θ range between 5 and 120°, with a step size of 0.01°. For each sample, 10 repeated 2 hr-scans were performed and summed up for data analysis, corresponding to a total acquisition time of 20 h. XRD analyses were performed in the Earth and Environmental Sciences Department, Faculty of Science, University of Waterloo, Canada.

4.2.3. Superpave Mix Design

According to AASHTO R 30-2 (2006), a Superpave mix design was performed. After preparing the aggregate gradation based on the specification requirements, the heated aggregate was mixed with the hot asphalt binder to produce the asphalt mixtures. To ensure that the asphalt binder was absorbed by the aggregates and achieved short-term ageing, the prepared samples were maintained at the compaction temperature for 2 h. The compaction and mixing temperatures were 150 °C and 163 °C, respectively. A Superpave (Rainhart An InstroTek company, Cedar Park, TX, USA) gyratory compactor was used to compact the samples. To replace coarse Nag, untreated CRCA portions were added to mixes at various proportions ranging from 0% to 60%. A 30% fraction of treated CRCA with different treatments was also utilized and added to create treated-CRCA-containing mixtures. Subsequently, the asphalt mixtures were produced according to the required design procedure. Table 8 shows the designed HMA mixtures that included various CRCA proportions and their volumetric properties.

Table 8. Volumetric characteristics of asphalt mixes that included untreated and treated CRCA at different proportions.

Asphalt Mixture/CRCA Type	CRCA	OAC [3]	VMA [4]	VFA [5]	Va [6]	D_p [7]	G_{mb} [8]
Control mix	0.0	4.83	14.52	72.5	4.0	0.6	2.40
Untreated CRCA mix	30	5.12	14.0	71.4			2.38
Dual-treated [1] CRCA mix	30	5.03	14.0	71.3		0.7	2.39
Dual-treated [2] CRCA mix	30	4.87	14.52	72.3			2.38
Untreated CRCA mix	60	5.2	13.27	70.03		0.8	2.37
MTO specification limit	-	-	13 *	65–75	4.0	0.6–1.2	-

Table 8. *Cont.*

Asphalt Mixture/CRCA Type	**CRCA**	**OAC** [3]	**VMA** [4]	**VFA** [5]	**Va** [6]	$\mathbf{D_p}$ [7]	$\mathbf{G_{mb}}$ [8]
Characteristic unit	%	%	%	%	%	%	-

[1] Dual-treated (heat treatment at 300 °C and short-term mechanical treatment) CRCA; [2] dual-treated (weak acid and short-term mechanical treatment) CRCA; [3] optimum asphalt content (OAC); [4] voids in mineral aggregates (VMA); [5] voids filled with asphalt (VFA); [6] air voids (Va); [7] dust-to-binder ratio (D_p); [8] bulk specific gravity (G_{mb}); * minimum value.

4.2.4. Asphalt Performance Tests

Thermal Stress Restrain Specimen Test

Using a shearbox compactor (PReSBOX) (Hoskin Scientific, Oakville, ON, Canada), the specimen beams were compressed with dimensions of L 39 cm, H 15 cm, and W 13 cm. The compacted beams were then sawed to meet the required dimensions (25 cm, 5 cm, 5 cm) for the thermal stress restrain specimen test (TSRST), and their air voids were then further evaluated to be within the acceptable range of 7 ± 1% [70,71]. Prior to compaction, the loose mixes were treated with a short-term thermal treatment at a temperature of 135 °C for 4 h to match the effects of plant mixing and placement, as per AASHTO R 30-02 (2006). To achieve their conditioning, the specimens' temperatures were maintained at 5 °C for 6 h in the test chamber. The test was performed at a fixed cooling rate (−10 °C/h). At the same time, initial tensile stress was simultaneously applied to the tested samples to prevent their contraction by restoring the specimen's initial length.

Indirect Tensile Strength (ITS) and Moisture Sensitivity Test

The ITS was measured for mixes containing raw and treated CRCA using a variety of processing techniques according to the AASHTO T-283 standard. To compress the mixes, a Superpave gyratory compactor was employed to obtain specimens with an approximate height of 95 mm with air void values of about 7%. The compressed specimens were categorized into two primary categories, each consisting of three samples: conditioned and unconditioned strengths. The test temperature was set at 25 °C, and the loading rate was 50 mm/min. Meanwhile, the remaining samples, which included unconditioned samples, were subjected to moisture conditioning. The initial conditioning step involved attaining a saturation level for the samples ranging from 70% to 80%. The samples were frozen at −18 ± 3 °C for at least 16 h. The specimens were subsequently submerged in a heated water immersion at 60 °C for a duration of 24 h. Following that, the specimens were placed in a water bath maintained at 25 °C for a duration of 2 h prior to their preparation for analysis. Determining the tensile strength ratio (TSR) involved the division of the value of conditioned strength by the unconditioned strength value. The TSR value, as directed by OPSS 1151 (2007), is expected to exceed 80%. Therefore, the TSR values must exceed the mentioned value to achieve successful values. The values of ITS and TSR are computed by applying the subsequent formulas:

$$\mathrm{ITS} = \frac{2000 \times \mathrm{P}}{\pi \times \mathrm{t} \times \mathrm{D}} \tag{7}$$

where ITS represents the value of indirect tensile strength in kPa; P stands for the maximum load applied in Newtons (N); t denotes the sample thickness before performing the test measured in mm; D stands for the sample diameter measured in mm; and π refers to the value of Pi (3.14159).

$$\mathrm{TSR} = \frac{\mathrm{ITS_{con.}}}{\mathrm{ITS_{uncon.}}} \times 100\% \tag{8}$$

where TSR stands for the tensile strength ratio; $ITS_{con.}$ is the tensile strength of the conditioned sample; and $ITS_{uncon.}$ is the tensile strength of an unconditioned sample.

Hamburg Wheel Rut Test

It is well known that pavement rutting is a serious type of asphalt road distress. This can compromise the safety of the road as well as the quality of the ride. This is completely true when the rutting depth reaches critical values [59,72]. In this research, the Hamburg Wheel Rut Tester (HWRT) was employed to evaluate the asphalt mixtures' rutting resistance based on the standard AASHTO T 324-04. The HWRT test application typically simulates the impacts of vertical compression and horizontal compaction generated by a wheel running over the asphalt pavement [49]. The experimental test was carried out in four replicates for each mix to obtain reliable results. The process of compacting every mixture was carried out utilizing a Superpave gyratory compactor; as a result, the compacted mixes achieved height values of 63 mm with air voids of about 7%. Under the influence of a solid steel wheel with an equivalent load of 705 ± 4.5 N, the samples were tested in a hot water bath at 50 °C for 10,000 cycles, approximately equivalent to 20,000 passes, or until the rutting depth reached 20 mm [59]. Linear variable differential transducers (LVDTs) were applied to evaluate the rutting depth under the influence of wheel loads.

5. Future Study Benefits

This research will promote the use of more RCA in asphalt mixes in Ontario, Canada, because its application is achievable using RCA treatment technologies. Regarding the conservation of natural resources, this research will help reduce the utilization of NA by urging the building and transportation sectors and private enterprises associated with these industries to switch from using NA to RCA. Moreover, the present research has the potential to reduce the burden placed on landfill sites caused by construction and demolition debris waste by providing other uses for RCA, which will have several favourable effects on both health and the environment. Lastly, this research will provide explicit instructions on the appropriate handling and optimal utilization of RCA in asphalt mixes in Ontario, Canada.

6. Conclusions

The impact of various treatment types on the ITZ of CRCA and its application in asphalt mixtures has been studied in this research. The following conclusions can be drawn based on the obtained experimental results:

- Heat treatment is a very successful method for improving the ITZ. Very high temperature (500 °C) heat treatment does not influence the ITZ; however, it is found that cracks form in the mortar of CRCA at high temperatures (500 °C), indicating a negative impact on mortar properties. Therefore, using temperatures between 300 °C and 350 °C is recommended.
- Treatment with acids appeared to be a successful method for enhancing the ITZ area. Nevertheless, according to the obtained outcomes, the treatment that used acetic acid, which is a weak acid, was more successful than the treatment that utilized HCl acid, which is a strong acid.
- Among various temperatures, heat treatment at 250 °C showed superior performance and significantly reduced the Ca/Si ratio, notably improving the ITZ. Nevertheless, at higher temperatures (350–500 °C), heat treatment negatively influences the atomic Ca/Si ratio.
- Acetic acid treatment significantly reduces the Ca/Si ratio for the ITZ, resulting in considerable improvement for the ITZ. HCl treatment appears to be effective for lowering the Ca/Si ratio and improving the ITZ.
- SEM analysis revealed that the untreated CRCA had a porous structure with a rough and uneven surface. On the other hand, the treated CRCA displayed reduced porosity and increased homogeneity, contingent upon the treatment used.
- Significant ITZ microstructure enhancement was obtained for various types of treatment. Nevertheless, better microstructure mainly includes increased C-S-H compounds, increased surface homogeneity, and a reduced Ca/Si ratio. In addition, there is a possibility of using an enhanced ITZ to increase the strength of CRCA.

- In terms of CSH compounds, the acetic acid treatment exhibited the best performance for increasing tobermorite and jennite compared to other treatment types. Nevertheless, there was a big difference between the two types of acid treatment when it came to the formation of jennite, indicating a specific sensitivity to the HCl acid environment.
- Compared to the control mix, 0% CRCA, it was noted that the average fracture temperature increased due to the impact of untreated CRCA addition on the thermal cracks at low temperatures. On the other hand, the fracture temperature was significantly reduced through the application of various treatment techniques.
- The dual treatment, which consisted of treatment with weak acid and short-term mechanical treatment, had less impact on the mix fracture temperature than the dual treatment that included heat treatment at a temperature of 300 °C and the same mechanical treatment.
- From the perspective of ITS, the findings showed that unconditioned and conditioned samples that included different untreated CRCA percentages had higher values than the control mix, registering a very effective performance for various mixes containing CRCA. The maximum ITS values were recorded for mixtures that included 30% untreated CRCA for unconditioned and conditioned samples.
- A reasonable improvement in the ITS values was recorded for mixtures that included 30% treated CRCA with different treatment techniques compared to those with 30% untreated CRCA. However, integrating heat and short-term mechanical treatment has the best performance for improving ITS values.
- The obtained results indicated that the TSR values of the mixes containing varying fractions of untreated CRCA exceeded the minimum threshold determined by MTO standards.
- The outcomes showed that the mixture that included 0% CRCA had a lower rutting resistance than the untreated CRCA mixes with different proportions of CRCA. This indicates that successfully improving resistance to permanent deformation can be accomplished by applying untreated CRCA.
- Significantly less permanent deformation was observed in the mix containing 30% CRCA that underwent dual treatment consisting of heat treatment and short-term mechanical treatment, compared to the mix containing the same proportion of untreated CRCA. This indicates a highly successful integration approach for improving the mixture's resistance to permanent deformation compared to other approaches.

Supplementary Materials: The following supporting information can be downloaded at https://www.mdpi.com/article/10.3390/recycling9030041/s1, Figure S1A: EDAX analysis of untreated CRCA. Figure S1B: EDAX analysis of treated CRCA with heat treatment at 250 °C. Figure S1C: EDAX analysis of treated CRCA with heat treatment at 350 °C. Figure S1D: EDAX analysis of treated CRCA with heat treatment at 500 °C. Figure S1E: EDAX analysis of treated CRCA with HCl acid. Figure S1F: EDAX analysis of treated CRCA with $C_2H_4O_2$ acid. Figure S2A: XRD analysis of untreated CRCA. Figure S2B: XRD analysis of treated CRCA with heat treatment at 250 °C. Figure S2C: XRD analysis of treated CRCA with heat treatment at 350 °C. Figure S2D: XRD analysis of treated CRCA with heat treatment at 500 °C. Figure S2E: XRD analysis of treated CRCA with HCl acid. Figure S2F: XRD analysis of treated CRCA with C_2H_4O acid.

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