DOI: https://doi.org/10.3329/jes.v14i2.71238

an international Journal

MICRO-CHARACTERIZATION OF RECYCLED CONCRETE AGGREGATE

Muhaiminul Islam Alim¹ and Kazi ABM Mohiuddin^{*1}

¹Department of Civil Engineering, Khulna University of Engineering & Technology, Khulna-9203, Bangladesh

Received: 14 December 2023 Accepted: 31 December 2023

ABSTRACT

Recycled concrete aggregate (RCA) is generated by crushing and processing concrete waste from demolition and construction activities, thereby reducing the environmental burden associated with disposing of concrete debris. The use of RCA in concrete production helps to alleviate the depletion of natural resources, as it serves as a substitute for traditional coarse and fine aggregates. This study identifies the microstructural aspects of RCA concrete, employing advanced techniques like scanning electron microscope (SEM) imaging with energydispersive spectroscopy (EDS) to analyze the microstructure and chemical composition of recycled concrete. The detailed microstructural analysis is anticipated to elucidate how the RCA content influences the interfacial transition zone, bond strength, and overall concrete performance. RCA exhibits a unique microstructure due to the presence of attached old mortar, which can influence the overall performance of RCA concrete. This study also explores the utilization of 100% RCA in preparing recycled concrete, comparing it with old and ordinary new concrete. The focus is on microstructural properties, hydration products, and the Interfacial Transition Zone (ITZ) between cement paste and aggregates. Samples were collected from a 47-year-old building, separating aggregates into different grades. Recycled coarse aggregates and recycled fine aggregates were obtained, and natural aggregates were collected. Concrete mixes for recycled and natural concrete were designed for a test strength of C30. Concrete cylinders were tested for compressive strength at 7, 28, and 90 days. Old concrete exhibited a dense microstructure with well-formed C-S-H gels, contributing to its superior strength. Recycled concrete, however, showed a wider and less dense ITZ, resulting in a more porous microstructure. EDS spectra confirmed C-S-H gel as the primary hydration product in all mixes, with the Ca/Si ratio varying, indicating complexity in hydrated product formation. The research highlights differences in microstructural and chemical characteristics among old concrete, ordinary new concrete, and recycled concrete. The wider ITZ and more porous microstructure in recycled concrete contribute to reduced strength. These findings provide insights into sustainable construction practices, promoting the use of recycled materials and addressing environmental challenges in the construction industry. As the construction industry continues to embrace sustainability, further research into the properties, performance, and best practices for incorporating RCA into concrete will play a pivotal role in advancing eco-friendly construction methodologies.

Keywords: Recycled concrete aggregate (RCA), Interface transition zone (ITZ), SEM-EDS.

1. INTRODUCTION

Since the beginning of the 21st century, with the rapid development of urbanization and industrialization, the construction industry has consumed large amounts of natural resources and produced large amounts of construction and demolition waste (Wang et al., 2020). The current era of globalization, marked by rapid economic growth, has spurred extensive infrastructure development worldwide. Concurrently, the renovation of old structures and buildings generates massive volumes of construction and demolition (C&D) waste in various countries. The rapid pace of urban development and the reconstruction of old city areas have generated a significant volume of construction waste. Inadequate disposal of this waste poses numerous environmental challenges and has adverse consequences. The impact on the environment is "Take 50% of raw materials from nature, consume 40% of total energy, and create 50% of total waste"; and construction became the industry with the largest impact on the environment (Kisku et al., 2017). Research shows that using RCA in concrete preparation not only satisfies the performance requirements of natural structural concrete but also solves the landfill shortage problem, which is consistent with the essence of sustainable development, namely, protecting the environment and the rapidly decreasing resources (Wang et al., 2020). In the pursuit of sustainability, C&D waste has become a focal point in the construction industry.

Today, the construction sector's emphasis on sustainability has resulted in the creation of durable concrete using industrial by-products, thereby reducing reliance on natural resources. The accumulation of waste concrete not

*Corresponding Author: kzmohiuddin@ce.kuet.ac.bd

https://www2.kuet.ac.bd/JES/

only occupies valuable land resources but also gives rise to significant environmental and social challenges. The most effective way to dispose of waste concrete is to recycle and reuse abandoned concrete, i.e., the application of recycled concrete (Chen, 2013). Construction waste contains mainly hardened paste, mortar, and concrete waste, and crushing construction waste into recycled aggregate as a natural aggregate substitute is one effective way to reduce construction waste (Kar et al., 2021; Ravathi & Chithra, 2022). Due to the limited availability of natural aggregates, recycled aggregate (RA) derived from construction and demolition (C&D) waste has emerged as a viable alternative in various engineering applications. Recent research has focused extensively on the utilization of RA in concrete.

Despite the promotion of recycled coarse aggregate (RCA) in construction practices over the last decade, there remain gaps in the utilization of both recycled coarse aggregate (RCA) and recycled fine aggregate (RFA) in construction, particularly concerning performance. Recycled aggregates inherently exhibit several drawbacks, including high porosity, significant internal cracks, and low apparent density. These characteristics contribute to a reduction in strength compared to concrete made with natural aggregates. Natural stone, old mortar, and their interfacial transition zone (ITZ) make up the majority of recycled aggregate, whose performance is inferior to natural aggregates' performance (Ahmed & Lim, 2020).

The microstructural properties of the concrete play a key role in the application of recycled concrete technology. This work focuses on the utilization of 100% recycled concrete aggregate for preparing recycled concrete. This study aimed to investigate the difference between old concrete, ordinary new concrete, and recycled concrete by using scanning electron microscopy (SEM) measurement, the microstructure of hydrated cement pastes, and the bonding characteristics of ITZ between cement paste and aggregate were also studied.

2. MATERIALS AND METHODS

2.1 Sample Collection and Preparation

Forty-seven-year-old concrete were collected from the old Khan Jahan Ali Hall Building at KUET campus, Khulna. Construction and demolition waste using visual inspection and hand sorting. The concrete of these buildings was made using brick chips. The old concrete was demolished, broken, and divided into two parts by sieving: recycled coarse aggregate and fine aggregate. In the production of recycled aggregates, a crucial step involves the separation of aggregates into different grades based on particle size. The RCA had a maximum aggregate size of 19 mm. The Recycled Coarse aggregates' particle size ranged from 19 to 4.75 mm. RCA derived from old concrete retains an old mortar layer, adding a layer of complexity to the aggregate and enhancing its bonding characteristics. Recycled fine aggregates (RFA) are obtained from the same parent building as the coarse aggregates. The recycled fine aggregates' particle size ranged from 0.075 to 4.75 mm. The washing process applied to recycled aggregates plays a pivotal role in enhancing their quality.

Properties	Materials					
	Recycled Coarse aggregates (RCA)	Recycled fine aggregates (RFA)	Natural Aggregate (Stone)	Natural Sand		
Unit weight (Kg/m ³)	1001	1329	1552	1632		
Fineness Modulus (FM)	-	2.99	-	3.22		
Specific gravity(SSD)	2.11	2.19	2.74	2.54		
Absorption capacity (%)	14.39	12.78	2.68	2.29		
Moisture content (%)	1.60	1.88	0.90	2.30		

 Table 1: Material properties of concrete

Table 2: Concrete mix design (weight basis)

		Concrete Properties		Weight (Quantity for each single batch)			gle batch)
Material	Target Strength	Target	Water	Cement	Water	Coarse	Fine
Properties	(MPa)	Sump	/Cement	(OPC)		aggregate	aggregate
Recycled	30	75 to 100	0.54	4.120	3.869	6.186	7.827
concrete							
Natural	30	75 to 100	0.54	4.120	2.410	9.591	7.639
concrete							

2.2 Concrete design and mixing, curing

Both Recycled Concrete and Natural Concrete were subjected to test strength of C30, and the water-cement ratio (W/B) was maintained at 0.54. The mix proportions for Recycled Aggregate concrete were 1:1:69:1.31. For ordinary natural concrete, they were 1:1.85:2.29. Concrete cylinders sized 150 mm in diameter and 300 mm in height, were cast for the evaluation of compressive strength and microstructural analysis at 7-, 28-, and 90-days following ASTM C 39/C 39M – 01 standards. Two groups, comprising a total of 10 samples, were prepared.

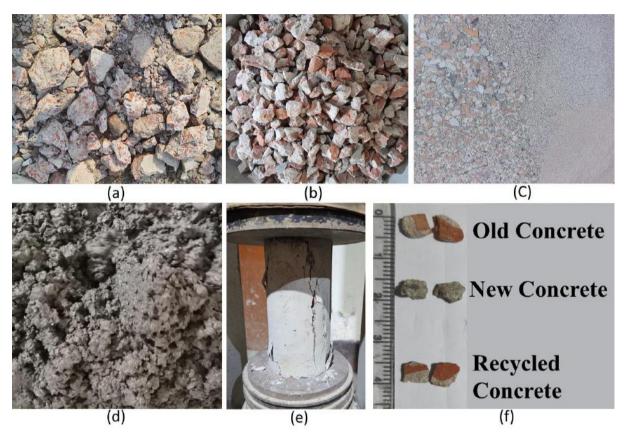


Figure 1: (a) Old demolished concrete, (b) Recycled Coarse Aggregate (c) Recycled Fine Aggregate (d)

Recycled Concrete Mixture, (e) Concrete Cylinder (Compression Test), (f) Sample for SEM Analysis

The concrete mixing process involved blending all solid ingredients, including cement, fine aggregate, and coarse aggregates. Water was then added to the mixtures with precise measurement. After mixing, the workability of the concrete was measured through the slump cone test. The steel moulds were filled in layers and subjected to 25 blows on each layer. Following a 24-hour period, the specimens were demolded and subsequently submerged in a water tank for 28 days. Compressive strength measurements were taken at 7, 28, and 90 days using a Compression Testing Machine (CTM). For microanalysis, samples were collected from the broken parts of the concrete cylinders.

2.3 SEM-EDS Analysis

The study used SEM-EDS to thoroughly analysis how Recycled Concrete Aggregate (RCA) affects the hydration products of the concrete matrix. This approach not only facilitated the examination of hydration changes but also allowed the measurement of the Interfacial Transition Zone's (ITZ) width within the concrete, serving as an indirect indicator of ITZ bond strength. By measuring the ITZ width across its length and determining the average width, the study provided valuable insights into the structural characteristics of each concrete variant.

The EDS study played a crucial role in identifying potential changes in hydration and compositional variations within the matrix, contributing to a comprehensive understanding of the concrete's structural and chemical characteristics.



Figure 2: (a) Gold plating upon the sample, (b) Sample placement on JEOL-JCM-7000 NeoScope[™] Benchtop SEM (c) Microstructure analysis through JEOL-JCM-7000 NeoScope[™] Benchtop SEM

SEM shows surface morphology and EDS shows the chemical composition of the old, new, and recycled aggregate concrete. The scanning electron microscope (SEM) model JEOL-JCM-7000 NeoScope™ Benchtop SEM was utilized to conduct a meticulous microscopic examination and analyze the elemental chemical composition of the samples. Using a distinctive lanthanum hexaboride filament as its electron source, this apparatus is equipped with secondary and backscattered electron detectors for image generation and an microanalysis system facilitated by EDS. Notably, the SEM can operate in variable pressure mode with low vacuum, eliminating the necessity for metallization when observing non-conducting samples. To optimize image resolution, high-vacuum conditions and gold-coated samples were employed in the experimentation. Utilizing a precision cutting saw, the concrete samples underwent meticulous sectioning, with each cut extracted from the central region of standard cylindrical specimens. The dimensions of the samples earmarked for SEM analysis measure approximately 6x5x4 mm.

3. RESULTS AND DISCUSSION

3.1 Concrete mix microstructure

This research aimed to discern the potential effects of integrating recycled aggregate on the formation and morphology of various hydration products within the concrete, including the Interfacial Transition Zone (ITZ) between the paste and the new aggregate, conducted through scanning electron microscopy (SEM) study. Through microanalyses, the chemical composition of these products was identified, with clear indications of the presence of Calcium Hydroxide (C-H) and Ettringite. CH is identified by its elongated crystalline form with well-defined edges, which was notably present on the aggregate surface.

Through the microscopic images presented in Figs. 3, 4, 5, and 6 capturing the interfacial transition zone (ITZ) between aggregates and cement pastes, the findings underscore substantial disparities in the microstructure of cement paste among old concrete, ordinary new concrete, and recycled concrete. This underscores the intricate relationship between material microstructure and its mechanical properties, providing valuable insights for further understanding and enhancing the performance of diverse concrete formulations. Furthermore, the SEM images Fig. 3(b), 4(a), and 6(a) showcased the measured ITZ width, offering a visual representation of the bond strength variations. Complementing these images, EDS spectrums were incorporated to visualize the distribution of various phases within the ITZ.

Fig. 3(a), 4(b), and 6(b) provide insight into the typical structure of Ettringite in new, old, and recycled concrete, highlighting its limited presence compared to other formations like Calcium Silicate Hydrate (C-S-H) and Portlandite (C-H). The distribution of elements in the paste, aggregates, and ITZ is depicted in another figure, showcasing consistency with the minerals' composition. Notably, elements were distributed following the principal components like Calcium Silicate Hydrate (C-S-H) and Portlandite (C-H) they contained and emphasizing the intricate chemical interplay within both natural and recycled concrete.

3.1.1 Old Concrete

The microstructure of old concrete is denser compared with that of ordinary new concrete and Recycled concrete. Old concrete has a massive amount of hydrated Calcium silicate hydrate (C-S-H) gel, Portlandite (C-H), and micro-particles that contribute to the generation of new C-S-H gel. Examining Fig. 4 reveals that the

cement paste microstructure in the old concrete specimens is quite compact, dense, uniform, and filled with hydration products. Old concrete exhibits superior density and smoothness on cement paste surfaces compared to both standard new concrete and recycled concrete.

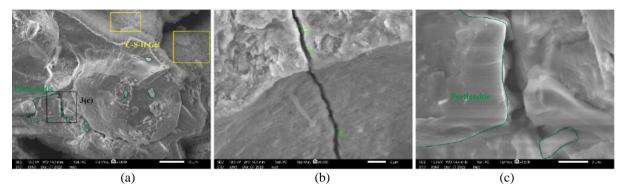


Figure 3: SEM images of (a) Microstructure of Old Concrete interface showing Calcium Silicate Hydrate (C-S-H) gel, Portlandtie (C-H) crystals (b) Old Interfacial Transition Zone (ITZ), (c) Old concrete showing Portlandtie (C-H) crystals.

Throughout the years, the efficient hydration response of cementitious composites, combined with the filling and sealing of diverse hydrates, resulted in a noteworthy decrease in both pore and micro-crack sizes within the matrix. The presence of numerous well-formed C-S-H gels played a crucial role. These gels formed a closely knit envelope around various hydration products and solute particles, establishing a dense and continuous phase. In old concrete specimens, the observation of micro-cracks and dissolute particles, was extremely challenging, except for a small pores zone. The well-developed C-S-H gels and their interlocking surfaces established a strong bond within the cement paste. The primary route of crack propagation occurs through the interfacial transition zone (ITZ), leading to the failure of the cement paste-aggregate binding. While the primary propagation of cracks occurs through the ITZ, between the cement paste and aggregate, secondary cracks are also evident within the cement paste itself. Cracks traverse the interfacial transition zone (ITZ) and extend from one aggregate to the next, choosing the most direct route. In the analyzed area, the maximum crack size is similar, the crack density is comparatively lower. The majority of these cracks are linked to the interface between the cement paste and aggregates, highlighting the structural intricacies. Within the examined region, a limited number of pores are evident.

3.1.2 Ordinary New Concrete

Old concrete exhibits a denser microstructure in comparison to typical new concrete, and the binding interface between natural aggregate and new paste surpasses that of the interface between recycled concrete aggregate and new paste. Consequently, ordinary new concrete demonstrates greater nucleation action and a more pronounced micro-aggregate filling effect than recycled concrete.

The microscopic analysis of ordinary new concrete reveals notable distinctions between old concrete and recycled concrete. In the case of the fresh material, the primary hydration products in the cement paste are Calcium Silicate Hydrate (C-S-H) gel and Portlandite (C-H) plates. However, certain regions of this hydrated cement paste exhibit varying densities, rough surfaces, and internal structures with voids, depressions, and irregular cracks, potentially diminishing the cement paste's strength. Fig. 4(b) illustrates that the Interfacial Transition Zone (ITZ) in the specimen is considerably larger compared to the old concrete. The ITZ between aggregates and cement paste displays reduced pore size and crack width due to adequate cement material hydration. As seen in Fig. 4(b), the gel components in the interfacial zone exhibit greater completeness and continuity compared to recycled concrete specimens. The hardening shrinkage of the cement paste triggers internal stress within the Interfacial Transition Zone (ITZ), leading to a separation between the aggregate and the cement paste. As a result, the gel layer in the ITZ maintains a relatively distinct spacing, adversely affecting the bond strength between the aggregate and the cement paste (He et al., 2020).

Fig. 4(a) illustrates a moderately compact cement paste in new concrete. Crack propagation mechanisms are identified in the examined region, primarily occurring through the Interfacial Transition Zone (ITZ) and, secondarily, within the cement paste. The average crack size measured in the analyzed area of new concrete is $8.887~\mu m$. Crack propagation takes place through both the ITZ and the cement paste in the fractures between raw aggregates. The maximum measured crack size reaches $12.70~\mu m$.

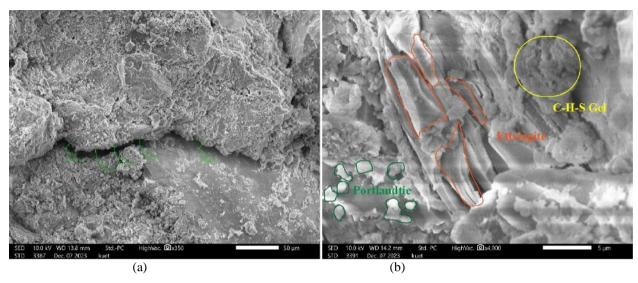


Figure 4: SEM images of Microstructure of Ordinary New Concrete interface (a) New Interfacial Transition Zone (ITZ), (b) Ordinary New concrete showing Calcium Silicate Hydrate (C-S-H) gel, Portlandtie (C-H) crystals and Ettringite (Aft) crystals.

3.1.3 Recycled Concrete

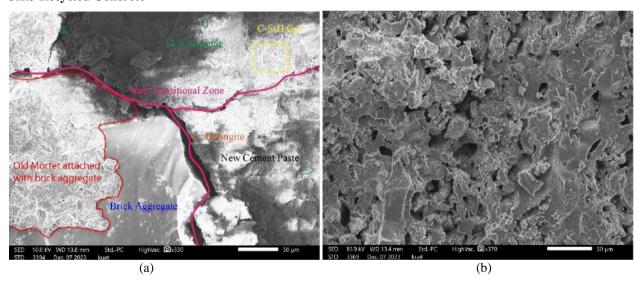


Figure 5: SEM images of Microstructure of Recycled Concrete interface (a) Interfacial Transition Zone (ITZ), (b) Pores or voids in Recycled Concrete Surface

The SEM analysis of recycled concrete exposes prominent pores or voids and inadequately formed interfacial zones within the sample structure. These voids introduce porosity, diminishing the overall compactness of the concrete structure. The increased recycled concrete aggregate content further contributes to void formation in the concrete. These porous zones diminish the particle-paste bond, resulting in a substantial reduction in concrete strength, as evident from the obtained results showcasing the concrete's weakened performance. The structure of recycled concrete appears notably distinct from that of new and old concrete, exhibiting a less dense composition. As a result, a higher occurrence of cracks is evident in the propagation through the cement paste compared to the incidence observed in both new and old concrete. The presence of two distinct concrete pastes contributes to cracks induced by the shrinkage of both the old and new cement pastes.

The structure of hydration products in recycled concrete, as depicted in Fig. 5, appears notably loose, and porous, and exhibits lower strength. The products occupy more space, facilitating the propagation of C-S-H gel, along with the presence of numerous Ca(OH)₂ and Ettringite (Aft) crystals. The recycled concrete displays larger Ca(OH)₂ and Ettringite (Aft) crystals, likely attributed to the larger pores in the recycled aggregate,

providing ample space for the growth of these crystals. In the new paste, recycled fine aggregate particles are enveloped by C-S-H gels, and the interface between a recycled fine aggregate particle and the new paste is dense, highlighting its nucleation action and micro aggregate filling effect. Upon contact with water, concrete ingredients undergo a process where highly mobile ions, such as Ca^{2+} , Al^{3+} , and $SO4^{2-}$, rapidly precipitate into less stable products like $Ca(OH)_2$ and Ettringite near the Interfacial Transition Zone (ITZ). Conversely, low-mobility ions, including silicate and ferrite, form hydration products in the proximity of the initial site (Behera et al., 2019).

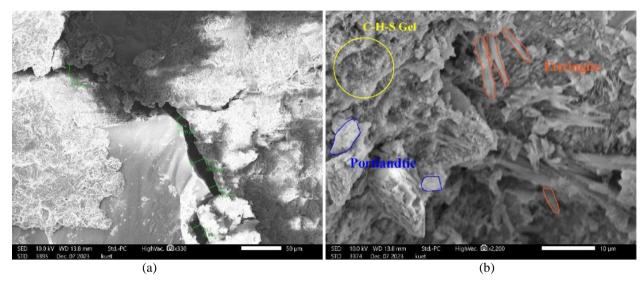


Figure 6: SEM images of the Microstructure of Recycled Concrete interface (a) Interfacial Transition Zone (ITZ)(b) Recycled Concrete showing Calcium Silicate Hydrate (C-S-H) gel, Portlandtie (C-H) crystals and Ettringite (Aft) crystals.

The internal structure of the hydrated cement paste in the recycled concrete aggregate specimen appeared less compact, characterized by numerous fluffy and snowflake crystals. Figure 6(a) illustrates the presence of abundant hydration products and solute particles surrounding the aggregates in the recycled concrete specimen. However, these hydration products exhibited numerous pores, failing to form a cohesive and continuous gel block. Notably, the interfacial transition zone (ITZ) displayed evident holes and wide cracks, contributing to its loose structure. The bond at the interface between brick aggregate and old paste surpassed that between recycled aggregate and new paste.

The presence of recycled concrete aggregate impeded the development of denser hydration products, primarily due to increased water absorption. The matrices of the resultant concrete mixes revealed a porous microstructure, encompassing both hydrated and non-hydrated products, along with dust from crushed aggregate. Notably, within these matrices, indications of partially reacted FA were present, displaying both large and small fibrous-like structures, which contributed to the formation of a less dense matrix (Behera et al., 2019). The incomplete hydrolysis of FA microspheres, attributed to variations in their chemical and physical structure, further hindered the achievement of full strength in the mixes. The presence of recycled fine aggregate and incomplete hydration processes resulted in the formation of large-size pore structures.

3.2 Interfacial transition zone (ITZ)

The SEM results illustrating the interfacial transition zone of recycled concrete are presented in Figures 3(b), 4(a), and 6(a). The figures show the composition and Interfacial Transition Zone (ITZ) of three different types of concrete: old concrete, ordinary new concrete, and recycled concrete. In ordinary new concrete, the composition mainly consists of natural aggregate, new paste, and the ITZ between them. Old concrete, on the other hand, consists of aggregate, old paste, and the old ITZ between them. Recycled concrete contains brick aggregate, old mortar paste, the ITZ between them, new mortar, and the new ITZ between recycled aggregate and new paste. This information helps to understand the differences in composition and ITZ in these different types of concrete.

3.2.1 Interfacial Transition Zone (ITZ) of Recycled Aggregate

The cement paste in old concrete is irregular and relatively dense, but in recycled concrete, the dense old paste, as well as the pores surface of the new paste, can be observed. The Interfacial Transition Zone (ITZ) of old concrete is observed to be intact and dense, attributed to the well-compacted hydration product, as illustrated in Figure 3(b). In the old concrete, the width of ITZ was found to be 533.17 nm. Comparing the ITZ in the old cement paste containing brick aggregate with the cement paste containing recycled aggregate, the ITZ of old concrete cement paste was much denser relative to cement paste containing recycled aggregate. The enhanced binding capacity between brick aggregate and old cement paste is attributed to the rough surface and irregular shape of the brick aggregate, thereby promoting favorable Interfacial Transition Zone (ITZ) properties. Additionally, active components in old concrete facilitate secondary hydration reactions, further benefiting the ITZ performance. In contrast, the ITZ of recycled concrete exhibits a wider interface between Recycled Concrete Aggregate (RCA) and new paste compared to old concrete and ordinary new concrete. This is attributed to the presence of massive porous hardened mortar and initial flaws in the recycled concrete waste, resulting in a relatively loose ITZ structure(H. Wu et al., 2023).

3.2.2 Interfacial Transition Zone (ITZ) of New Concrete

The interfacial transition zone (ITZ) in ordinary new concrete is less compact compared to that in old concrete, and the micro-cracks in the interface of ordinary new concrete are wider than those in old concrete (Chen, 2013). In Fig. 4(a), the measured crack size in the analyzed area of recycled concrete was $8.887~\mu m$. The largest fissure size observed in the analyzed region of Recycled Concrete Aggregate (RCA) is $12.70~\mu m$. Furthermore, the new interfacial transition zone of recycled concrete is considerably broader than that of ordinary new concrete.

3.3.3 Interfacial Transition Zone (ITZ) of Recycled Concrete

Recycled concrete is distinguished by the presence of the interfacial transition zone (ITZ) between recycled concrete aggregate and cement pastes, which plays a pivotal role in influencing its macroscopic mechanical properties. The microstructure of the Interfacial Transition Zone (ITZ) in recycled concrete, created by the interaction of recycled aggregate and new cement paste, is notably intricate, showcasing the presence of multiple ITZs. This complexity arises from the presence of numerous narrow ITZs, surrounded by both the old cement paste and the new cement paste enveloping the aggregate. This intricate structure contributes to the expansion of the new ITZ, exerting a discernible impact on the ultimate hardened properties of recycled concrete.

Recycled concrete consists of recycled coarse aggregate (RCA) and recycled fine aggregate (RFA), surrounded by a matrix of both hydrated and unhydrated cement paste, accompanied by fine crushed aggregate dust. Nevertheless, the combination of Recycled Concrete Aggregate (RCA) and Recycled Fine Aggregate (RFA) in the mixes led to the formation of a distinctive Interfacial Transition Zone (ITZ) characterized by noticeable porosity and a discernible gap between the matrix and the aggregate zone within the ITZ. This phenomenon may be attributed to the heightened water absorption characteristics of RFA, leading to micro-bleeding of water near the aggregate surface. Consequently, the accumulated water in the ITZ either evaporates or participates in the hydration process, creating a gap. The increased micro porosity near the ITZ in RCA remains unfilled by hydration products. Loose particles from a layer lead to a less dense volume of hydration product formation. As a consequence, the space between the recycled aggregate and the paste phase is not filled by the C-S-H hydration product. This leads to a less dense and intact Interfacial Transition Zone (ITZ) in the resulting concrete. Moreover, defects in the orientation of partially hydrated cement particles or the packing of unreacted Recycled Fine Aggregate (RFA) particles near the ITZ may contribute to the widening of the ITZ (Behera et al., 2019).

The cracks in RCA result from the propagation path of the damage mechanism detected in the sample. Two explanations for the presence of cracks in RCA are the shrinkage of the new paste and the existence of previous cracks in old concrete. From Fig. 6(a), in the analyzed area, the measured crack size in recycled concrete was $12.824 \, \mu m$, while the maximum measured fissure size in RCA was $18.28 \, \mu m$.

3.4 Energy Dispersive Spectroscopy (EDS) Analysis

The atomic distribution of hydration products/phases in the matrix and aggregate was elucidated through the EDS spectrum. The EDS study aimed to detect any alterations in hydration or compositional variations in the matrix. Figures 7, 8, and 9 present SEM images of the matrices of different concrete samples, accompanied by the corresponding EDS spectra. The Interfacial Transition Zone (ITZ) consistently exhibited prominent peaks

corresponding to Ca, Si, and O. Microstructure analysis revealed a more porous microstructure in the ITZ, directly impacting the mechanical properties of concrete. The distribution of calcium, alumina, and silicate ions across the matrix provides evidence that the hydration product in all mixes is the C-S-H gel (Medina et al., 2012).

The matrix of the control concrete from the old samples, as depicted in Figure 7, exhibits a compact, dense structure thoroughly filled with hydration products. This contributes to its superior strength in comparison to the other mixtures. Figure 9 presents micrographs showcasing the interface between the new cement paste and the recycled aggregate paste, accompanied by microanalyses highlighting the various structures present. The Interfacial Transition Zone (ITZ) of the old concrete is narrower, more compact, and less porous when compared to the interface of recycled concrete. This leads to enhanced integration of the brick aggregate with the concrete matrix, predominantly consisting of calcium-silicate-hydrate (C-S-H). The less porous and less continuous Interfacial Transition Zone (ITZ) in old concrete renders it less permeable to aggressive agents, encompassing both chemical and environmental factors.

In the microanalyses corresponding to Figure 7, the primary elements identified were Silicon (Si), Aluminum (Al), Calcium (Ca), and Sulfur (S). The presence of Silicon (Si) suggests the possibility of thaumasite, a formation similar in morphology to ettringite but distinguished by its unique chemical composition, incorporating silica instead of alumina. The presence of silica peaks in both ettringite formations can be attributed to challenges in the paste and does not actively participate in the chemical reactions taking place during cement hydration.

The characterization of the reaction product was carried out by assessing the Ca/Si ratio, with the results representing average values obtained from various points within the matrix. The rise in the Ca/Si ratio indicates the existence of C-H structures and delayed hydrolysis of cement particles within the matrices. The fluctuations in the lime-to-silica (Ca/Si) ratio across the matrix underscore the complexity of the hydrated products formed in

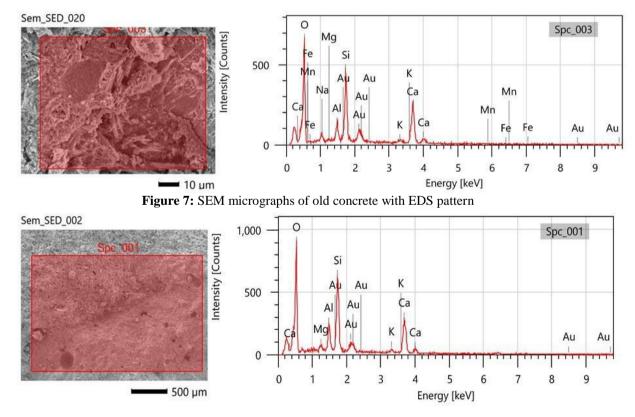


Figure 8: SEM micrographs of ordinary new concrete with EDS pattern

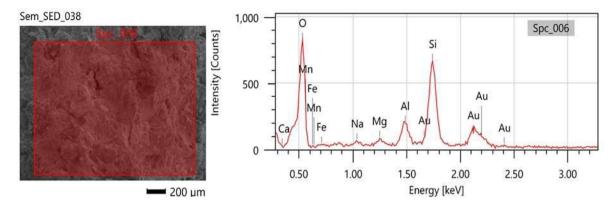


Figure 9: SEM micrographs of recycled concrete with EDS pattern

Table 3: Energy Dispersive Spectroscopy (EDS) of Different Element on concrete according to %Mass and % Atom.

Element	Line -	Old Concrete		Ordinary New Concrete		Recycled Aggregate	
	Line	Mass%	Atom%	Mass%	Atom%	Mass%	Atom%
О	K	41.40 ± 1.76	64.30±2.73	42.91±1.57	64.63±2.37	37.42±1.40	61.66±2.31
Na	K	1.28 ± 0.25	1.38 ± 0.27	-	-	0.73 ± 0.16	0.84 ± 0.19
Mg	K	0.22 ± 0.11	0.23 ± 0.12	0.83 ± 0.17	0.82 ± 017	0.82 ± 0.17	0.89 ± 0.19
Al	K	3.11 ± 0.38	2.86 ± 0.35	4.42 ± 0.40	3.95 ± 0.35	4.05 ± 0.38	3.96 ± 0.37
Si	K	12.71±0.76	11.24 ± 0.67	14.96 ± 0.73	12.84 ± 0.62	15.72 ± 0.74	14.75 ± 0.70
K	K	1.22 ± 0.36	0.78 ± 0.23	1.91±0.39	1.18 ± 0.24	1.09 ± 0.30	0.75 ± 0.20
Ca	K	27.52 ± 1.77	17.06±1.10	25.68 ± 1.52	15.44 ± 0.91	19.91±1.33	13.10 ± 0.88
Mn	K	0.29 ± 0.52	0.13 ± 0.24	-	-	0.97 ± 0.68	0.47 ± 0.33
Fe	K	1.48 ± 1.09	0.66 ± 0.49	-	-	3.03 ± 1.34	1.43 ± 0.63
Au	K	10.78 ± 1.23	1.36 ± 0.15	9.28 ± 1.01	1.14 ± 0.12	16.25 ± 1.32	2.17 ± 0.18
Total	•	100	100	100	100	100	100
Fitting Ratio		0.1488		0.148		0.1637	

the matrices of different mixes. The Ca/Si ratio for old concrete was determined to be 2.17. In contrast, the ratios were 1.64 and 1.27 for ordinary new concrete and recycled aggregate, respectively. The decrease in the Ca/Si ratio with the incorporation of Recycled Concrete Aggregate (RCA) and Recycled Fine Aggregate (RFA) aligns with a decrease in compressive strength. The matrices of recycled concrete exhibited relatively higher porosity compared to both old concrete and ordinary new concrete.

4. CONCLUSIONS

The research focuses on the utilization of 100% recycled concrete aggregate (RCA) in preparing recycled concrete and aims to investigate the differences between old concrete, ordinary new concrete, and recycled concrete. The study employs SEM-EDS to analyze the microstructure, hydration products, and bonding characteristics of the Interfacial Transition Zone (ITZ) between cement paste and aggregates. SEM analysis reveals substantial differences in microstructure among old concrete, ordinary new concrete, and recycled concrete. Old concrete exhibits a denser microstructure with well-formed Calcium Silicate Hydrate (C-S-H) gels, contributing to its high strength. The ITZ in recycled concrete is wider and less dense than old and ordinary new concrete, affecting its microstructural properties. The presence of recycled aggregate impedes the development of less dense hydration products, resulting in a more porous microstructure. EDS spectra confirm the presence of Ca, Si, and O across the ITZ, indicating C-S-H gel as the hydration product in all mixes. Ca/Si ratio variations across matrices suggest complexity in hydrated product formation, with recycled concrete matrices being more porous. Cracks in recycled concrete are attributed to the shrinkage of new paste and existing cracks in old concrete. The crack size in recycled concrete is larger than in ordinary concrete. The research provides valuable insights into the microstructural and chemical characteristics of old concrete, ordinary new concrete, and recycled concrete. Understanding the differences in their properties contributes to the ongoing efforts for sustainable construction practices by promoting the use of recycled materials.

ACKNOWLEDGEMENT

The authors gratefully acknowledge the support of the Center of Excellence and Shear Research Facilities of KUET for SEM-EDS analysis.

REFERENCES

- Ahmed, W., & Lim, C. W. (2020). Production of Sustainable and Structural Fiber Reinforced Recycled Aggregate Concrete with Improved Fracture Properties: A Review. *Journal of Cleaner Production*, 279, 123832.
- Behera, M., Minocha, A. K., & Bhattacharyya, S. K. (2019). Flow behavior, microstructure, strength and shrinkage properties of self-compacting concrete incorporating recycled fine aggregate. *Construction and Building Materials*, 228, 116819.
- Chen, B. (2013). Basic mechanical properties and microstructural analysis of recycled concrete. *Journal of Wuhan University of Technology-Mater. Sci. Ed.*, 28(1), 104–109.
- Evangelista, L., & Guedes, M. (2019). Microstructural studies on recycled aggregate concrete. In *New Trends in Eco-efficient and Recycled Concrete* (pp. 425–451). Elsevier.
- He, W., Kong, X., Fu, Y., Zhou, C., & Zheng, Z. (2020). Experimental investigation on the mechanical properties and microstructure of hybrid fiber reinforced recycled aggregate concrete. *Construction and Building Materials*, 261, 120488.
- Kar, D., Giri, J. P., Panda, M., & Chattaraj, U. (2021). Investigations on Stone Matrix Asphalt Mixes Containing Recycled Concrete Aggregate Treated with Nanosilica. *Journal of Materials in Civil Engineering*, 33, 04021228.
- Kong, D., Lei, T., Zheng, J., Ma, C., Jiang, J., & Jiang, J. (2010). Effect and mechanism of surface-coating pozzalanics materials around aggregate on properties and ITZ microstructure of recycled aggregate concrete. *Construction and Building Materials*, 24(5), 701–708.
- Medina, C., Frías, M., & Sánchez De Rojas, M. I. (2012). Microstructure and properties of recycled concretes using ceramic sanitary ware industry waste as coarse aggregate. *Construction and Building Materials*, 31, 112–118.
- Ravathi, M. C., & Chithra, R. (2022). Experimental and Analytical Evaluation of the Mechanical Properties of High-Strength Self-Curing Concrete with Recycled Fine Aggregates. *Journal of Materials in Civil Engineering*, 34.
- Ruben, J. S., Sophia, M., & Raja, M. A. (2022). Mechanical and Durability Properties of Self Compacting Concrete Containing Treated Recycled Coarse Aggregates and Acetic Acid-Metakaolin Slurry. *Iranian Journal of Science and Technology, Transactions of Civil Engineering*, 46(4), 2933–2959.
- Thomas, C., De Brito, J., Cimentada, A., & Sainz-Aja, J. A. (2020). Macro- and micro- properties of multi-recycled aggregate concrete. *Journal of Cleaner Production*, 245, 118843.
- Verma, A., Sarath Babu, V., & Arunachalam, S. (2021). Influence of mixing approaches on strength and durability properties of treated recycled aggregate concrete. *Structural Concrete*, 22(S1).
- Wang, R., Yu, N., & Li, Y. (2020). Methods for improving the microstructure of recycled concrete aggregate: A review. *Construction and Building Materials*, 242, 118164.
- Wang, X., Yang, X., Ren, J., Han, N., & Xing, F. (2021). A novel treatment method for recycled aggregate and the mechanical properties of recycled aggregate concrete. *Journal of Materials Research and Technology*, 10, 1389–1401.
- Wu, H., Yang, D., Wang, C., & Ma, Z. (2023). Microstructural and Macroperformance of Recycled Mortar with High-Quality Recycled Aggregate and Powder from High-Performance Concrete Waste. *Journal of Materials in Civil Engineering*, 35(3), 04022482.
- Wu, J., Zhang, Y., Zhu, P., Feng, J., & Hu, K. (2018). Mechanical Properties and ITZ Microstructure of Recycled Aggregate Concrete Using Carbonated Recycled Coarse Aggregate. *Journal of Wuhan University of Technology-Mater. Sci. Ed.*, 33(3), 648–653.

© 2023 JES. Journal of Engineering Science published by Faculty of Civil Engineering, Khulna University of Engineering & Technology. This is an open access article under the terms of the Creative Commons Attribution-Noncommercial-No Derivatives License, which permits use and distribution in any medium, provided the original work is properly cited, the use is non-commercial and no Modifications or adaptations are made.