**Research Article****Microanalysis of fly ash-based composite landfill liners**

Md. Mehedi Emon*, Aiman Al-Odaini and Dato Ir Jamaludin Bin Non

*Faculty of Engineering, Lincoln University College, Malaysia***ARTICLE INFO****Article History**

Received: 08 February 2026

Revised: 13 April 2026

Accepted: 26 July 2026

Keywords: Landfill liner material, Fly ash-based composite, SEM-EDS, X-ray diffraction, Pozzolanic reactions, Sustainable waste management.

ABSTRACT

The production of low-cost and eco-friendly landfill liner materials is necessary in areas where engineered leachate collection systems are not available. This article investigates the potential of locally available fly ash (FA), white clay (WC) and cement kiln dust (CKD) for preparing a composite landfill liner system with satisfactory mechanical and containment properties. Laboratory tests revealed that the designed FA:WC:CKD mixture had a compressive strength of 1.64 MPa, fines content of 71%, and specific gravity 2.42, suggesting its applicability for liners. Samples were analyzed by SEM-EDS (Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy), where the dense microstructure found was observed to be well sintered, and calcium-, silicate- and aluminosilicate-rich phases dominated. The crystalline products of pozzolanic and cementitious reactions were verified by X-ray Diffraction (XRD). The integrated mechanical, microstructural, and mineralogical properties indicate that the FA-WC-CKD composite is a potential low-cost medium for long-term leachate confinement in landfills.

Introduction

Safe containment of landfill leachate is a key environmental need, especially in developing areas where engineered landfills are rarely equipped with sophisticated leachate collection and treatment installations. In these conditions, landfill lining material is required to provide hydraulic stability, mechanical resistance, and chemical inertia over long time spans at a reasonable price in an eco-friendly way (Rowe, 2005; Daniel and Koerner, 2007).

According to Bouazza et al. (2017) and Rowe et al. (2017), low hydraulic conductivity and contaminant attenuation capacity make conventional clay-based barrier systems, e.g., compacted clay liners and geosynthetic clay liners (GCLs), a common component of landfill containment. However, their performance is very much dependent on the material properties and site-specific conditions, and the use of

large quantities of natural clay may raise issues of resource availability and sustainability. Therefore, alternative liner materials based on locally available natural resources and industrial by-products have attracted increasing attention. Among these, fly ash (FA), white clay (WC) and cement kiln dust (CKD) are of particular interest, as they are naturally abundant, inexpensive, and have desirable physicochemical properties (Yoon et al., 2009; Aziz et al., 2015).

Fly ash has reactive aluminosilicate phases to induce pozzolanic reactions, and CKD gives free lime and alkalis for cementitious bonding. White clay provides fine particles in appreciable quantity, which improves plasticity and reduces hydraulic conductivity. When mixed adequately, these materials can create a compact, impermeable

*Corresponding author: <mehedi.emon@gmail.com>



This work is licensed under a Creative Commons Attribution 4.0 International License.

composite viable for landfill liner applications (Edil et al., 2012).

This is because there is ever-increasing demand for high-performance and durable materials in civil, environmental, and industrial engineering, which has resulted in renewed interest in the study of material behavior at the microstructural or mineralogical scale. Material behavior, specifically the mechanical strength and durability of material (resistance to chemical attack), is largely controlled by its pore structure, bonding phases, and elemental composition, which form due to physico-chemical reactions within the materials as they are processed and used (Scrivener et al., 2018; Taylor, 1997).

Advanced characterization methods, including Scanning Electron Microscopy (SEM) combined with Energy Dispersive X-ray Spectroscopy (EDS) and X-ray Diffraction (XRD) contribute to an understanding of microstructural densification, elemental distribution, and crystalline phase evolution. The SEM allows observation of pore shape and particle bonding, whereas EDS provides local elemental compositions at the micro- to sub-microscale. XRD will complement this information by identifying crystalline phases contributing to mechanical strength and long-term chemical stability (Goldstein et al., 2018; Cullity and Stock, 2014).

The calcium-, silicate- and alumina-rich phases are particularly relevant for engineered materials in which they contribute to pozzolanic and cementitious reactions, generating denser matrices with lower permeability (Neville, 2011). The thermal stability, corrosion resistance and wear resistance of the samples, in both normal and severe environmental circumstances (and especially in the latter), can also be greatly improved by trace elements such as zirconium, tantalum and niobium (Lütjering and Williams, 2007).

This research explores the potential of utilizing a composite material that consists of FA, WC and CKD to formulate a cost-effective and environmentally-friendly landfill liner that can retain

leachate over extended periods in the absence of an incorporated leachate collection system. In this context, the present work focuses on a systematic analysis of microstructure features, element distribution, and crystalline phases in the material by using SEM–energy dispersive X-ray spectroscopy (EDS) and XRD methods. The results are discussed in terms of the mechanism of densification, durability, and chemical stability that support the material’s potential for high-performance engineering applications.

Methodology

Material Preparation and Laboratory Testing

FA, WC and CKD were locally obtained and characterized before mixing. Several mix ratios were experimentally established and tested in the laboratory to determine the best proportion for mechanical and hydraulic performance.

The test program included compressive strength testing for load-bearing capacity, Particle size distribution for gradation and fines content determination, specific gravity and the STAT needle test for liner suitability.

Based on these tests, the FA:WC:CKD combination with the best characteristics was chosen for additional microstructure study. The best combination obtained a maximum compressive strength (1.64 MPa), 71% fines, and specific gravity of 2.42, which satisfied critical requirements in order to design compacted liner material.

Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS)

The surface morphology, pore structure and the bonding state of the particles in the material were observed through Scanning Electron Microscopy (SEM), in a wide range of magnifications (from 30× to 10,000×). High-resolution micrographs were used to evaluate densification, pore connectivity, and reaction product presence.

Qualitative and quantitative elemental analysis of samples was performed by Energy Dispersive X-ray Spectroscopy (EDS) attached to the SEM. Elemental composition was determined at localized points

(Points 3-7), and spectral (Spectra 2-6) analyses were performed. The EDS spectra showed elemental peaks at characteristic X-ray energies, allowing determination of major, minor and trace elements. Atomic and weight percentages for these elements were measured to distinguish between light (e.g., C, O) and heavy (e.g., Ca, Zr, Ta, Nb) elements that have a greater impact on material behavior but lower atomic prevalence (Goldstein et al. 2018).

In SEM, analysis is conducted at a particular, small area on the surface sample using microanalytical techniques such as Energy-dispersive X-ray Spectroscopy (EDS or EDX). The spectrum is generally referred to as the resultant data from point analysis, which displays energy peaks of different elements in the areal analyzed. It plots X-ray intensity against energy, revealing the elemental composition of the substance.

X-ray Diffraction (XRD) Analysis

X-Ray Diffraction (XRD) measurements were

performed to identify the crystalline phases in the composite and monitor mineralogical changes associated with increased strength and chemical durability. Patterns were recorded as a function of 2θ , and characteristic diffraction peaks were studied to identify Calcium-silicate-hydrate (C-S-H), Calcium-aluminosilicate-hydrate (C-A-S-H), Carbonate, or oxide phases.

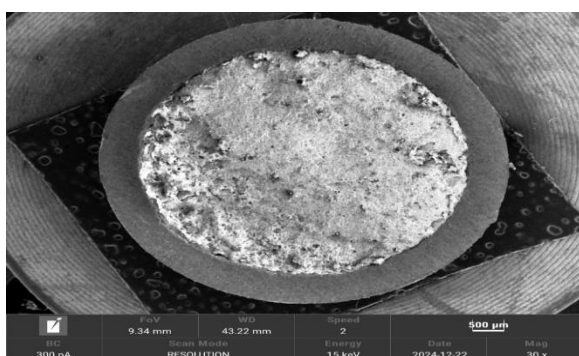
Despite of the fact that XRD is focused on bulk features (and not point-wise information), it is essential for discriminating reaction products identified by SEM–EDS analysis.

Integrating SEM–EDS and XRD provides a complete investigation of microstructural characteristics and mineralogical composition.

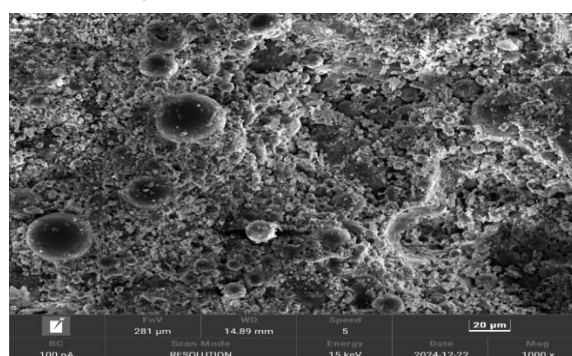
Data analysis

Microstructural Interpretation from SEM Images

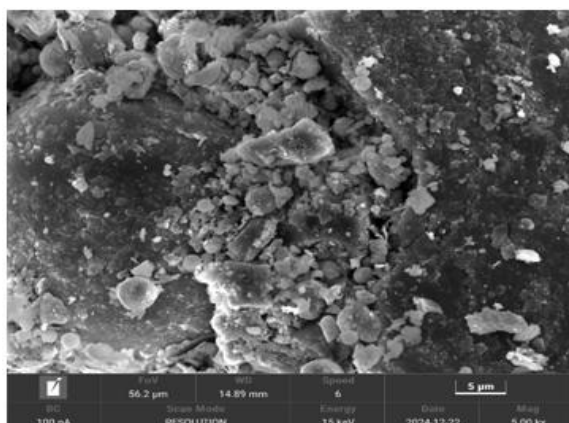
The SEM images shown in Fig. 1 were taken at different magnifications and locations to describe the



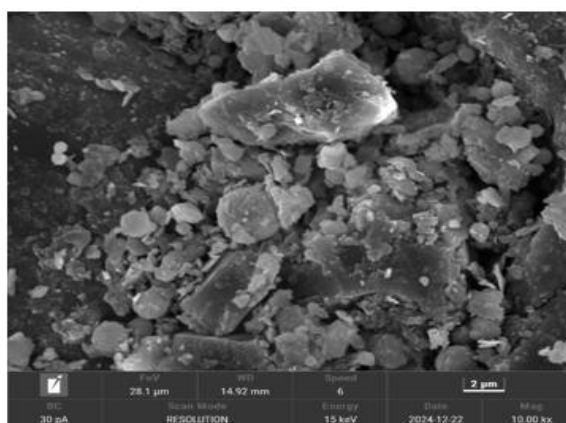
(a) 30X



(b) 1000X



(c) 5000X



(d) 10000X

Fig. 1. Electron Microscope Images of the Concerned Material at multiple magnifications.

microstructure evolution of the FA-WC-CKD composite. At lower magnifications ($30\times$ - $1000\times$; Fig. 1(a) and (b)), the material shows a generally homogeneous the composite matrix with no macro-cracks, implying good mixing and full compaction. The low percentage of large, connected voids at this scale also indicates favorable condition for low hydraulic conductivity.

At higher magnification ($5000\times$ - $10,000\times$; Fig. 1 (c), and (d)), the microstructure becomes denser, small particles fill intergranular porosity, and reaction products from continuous bonding phases. Agglomerated particle arrays connected by cementitious bridges suggest pozzolanic and hydrate reactions between fly ash aluminosilicates and calcium-rich phases mainly from CKD. The dense, interlocked microstructure is particularly important for landfill liner applications, as it increases packing density, reduces pore connectivity, and improves resistance to leachate migration.

Elemental Composition and Distribution (EDS Point Analysis)

The SEM images from **Points 3–7** are localised and show microstructural homogeneity in different areas (Fig. 2). Despite some textural differences, there are no zones of high pore contentions or poor bonding; there is no sign and the liner material appears to be performing uniformly.

The band positions indicated in each spectrum represent localized chemical environments in the framework (Fig.3). The similarity between the strong O, Ca, and C peaks in all point spectra indicates a chemically uniform matrix. Smaller differences in bandwidth width and peak intensity indicate microscale compositional variations, including (CKD hydration product) Calcium-rich zones, and FA-plus-WC-particles (Aluminosilicate-rich regions).

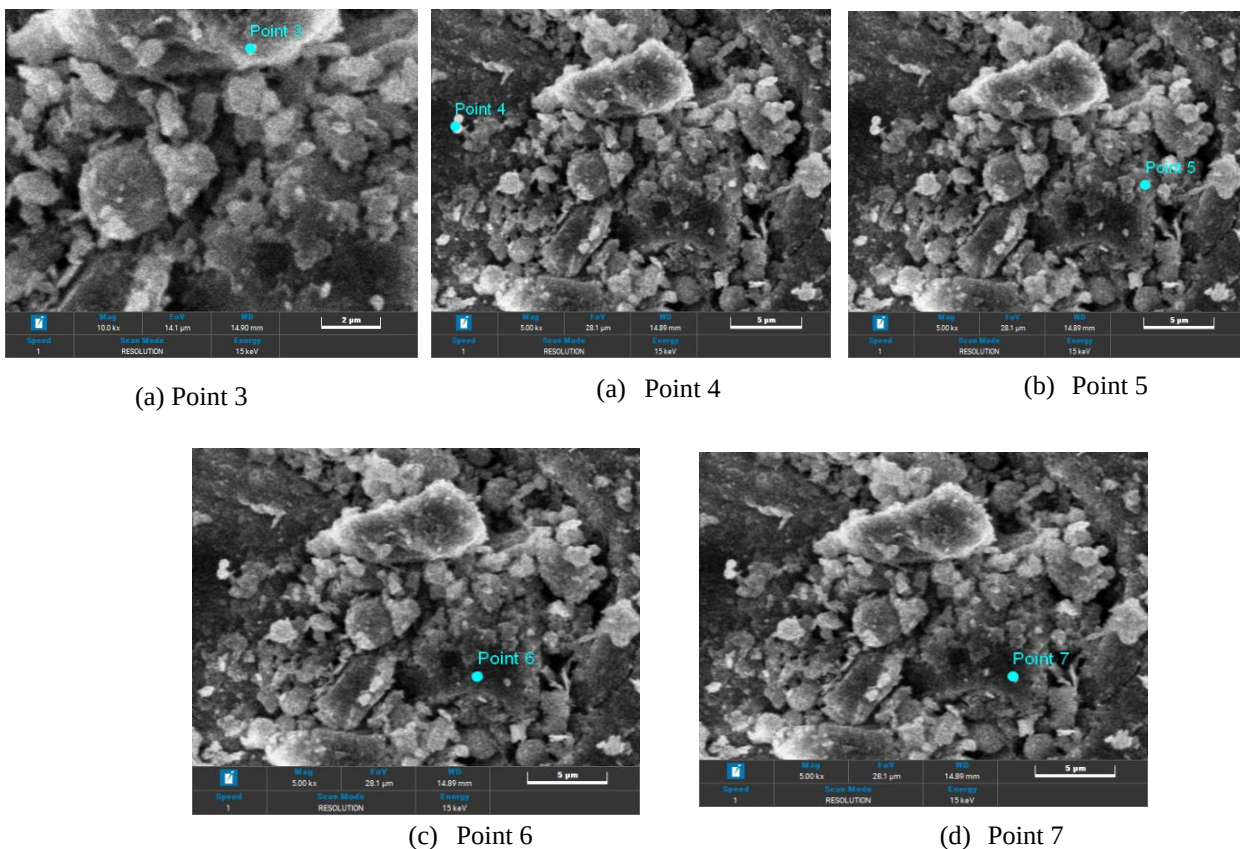
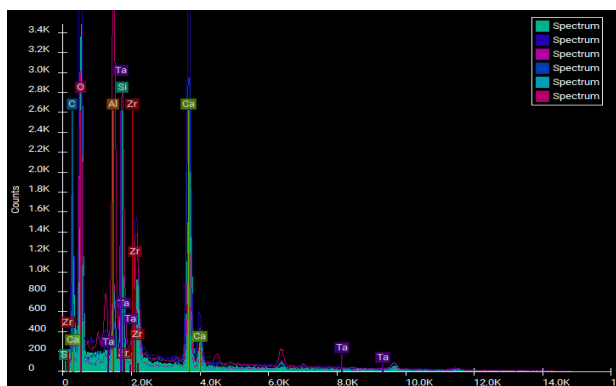
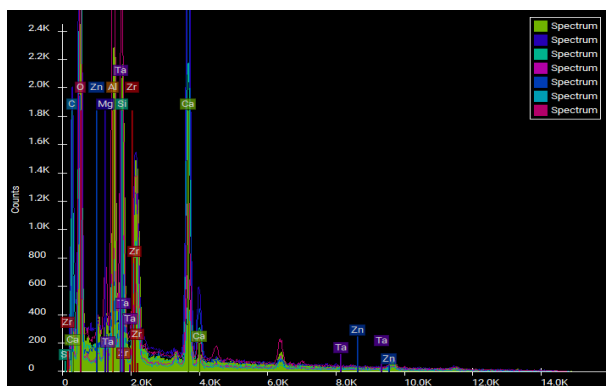


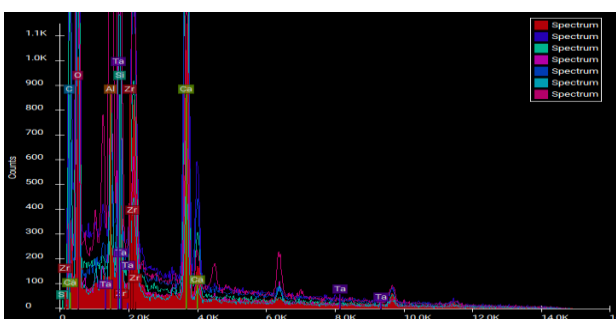
Fig. 2. Electron Microscope Images of the Composite Material at different points.



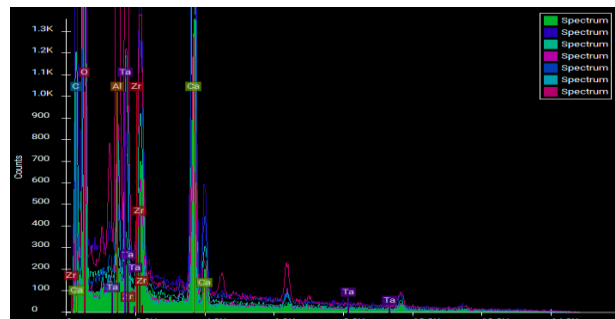
(a) Point 3



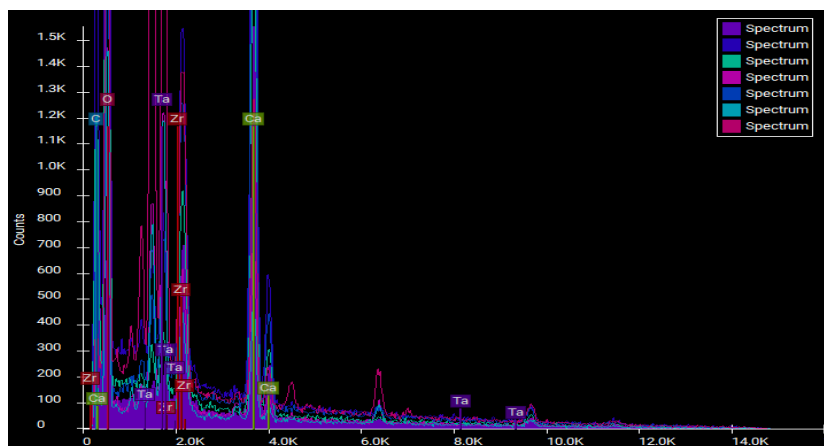
(b) Point 4



(b) Point 5



(b) Point 6



(b) Point 7

Fig. 3. Spectrum Bandwidth of the composite material at different points.

The EDS point analyses presented in Table 1 provide quantitative elemental data at specific microstructural sites. On the other hand, carbon, oxygen, and calcium randomly dominate in atomic values (average wt %) and weight values (average at calcium content ranges ~ 9-18% (atomic) and up to 34% (weight), demonstrating the predominance of CKD as a calcium donor. This calcium supply is necessary to initiate pozzolanic reactions with silicon and aluminum compounds in fly ash and primary clay. Similarly, silicon and aluminium, albeit at low concentration, support the development of the C-S-H and C-A-S-H gels, which are also well known for enhancing mechanical properties and reducing permeability.

Trace elements such as zirconium, tantalum, zinc and magnesium found at a number of locations have relatively low atomic percent but higher weight

percent values. Their distributed spatial profile suggests that they are not pure contaminants, but that the elements can also form a complex with the matrix. Although not being major strength contributors, these elements are associated with increased resistance to chemical attack and aggressive environments, which can be advantageous for the long-term performance of landfill lining.

Spectrum Analysis

The EDS spectra in Fig. 4 also support the point-analysis data by presenting roughly similar peak intensities of oxygen, carbon, and calcium across different spectra. The persistent occurrence of these predominant peaks reveals that the matrix exhibits chemical stability and compositional homogeneity.

Table 1. Percentage of Atomic and Weight in terms of inherit elements of the material at different points.

Element	Point 3		Point 4		Point 5		Point 6		Point 7	
	Atomic %	Weight %	Atomic %	Weight %	Atomic %	Weight %	Atomic %	Weight %	Atomic %	Weight %
Carbon	41.54%	28.85%	34.79%	20.21%	40.30%	22.81%	52.49%	33.64%	59.03%	40.05%
Oxygen	47.09%	43.57%	40.82%	31.59%	39.84%	30.04%	31.85%	27.19%	27.81%	25.13%
Magnesium	-	-	1.48%	1.74%	-	-	-	-	-	-
Aluminum	0.58%	0.91%	9.02%	11.77%	1.36%	1.73%	0.86%	1.23%	-	-
Silicon	0.62%	1.01%	6.90%	9.37%	1.13%	1.50%	-	-	-	-
Zinc	-	-	3.09%	5.99%	-	-	-	-	-	-
Calcium	9.46%	21.93%	0.97%	3.05%	14.39%	27.18%	13.14%	28.11%	11.93%	27.01%
Zirconium	0.70%	3.71%	2.17%	9.59%	2.03%	8.75%	1.30%	6.33%	0.92%	4.75%
Tantalum	0%	0.01%	0.77%	6.70%	0.94%	7.99%	0.36%	3.50%	0.30%	3.05%

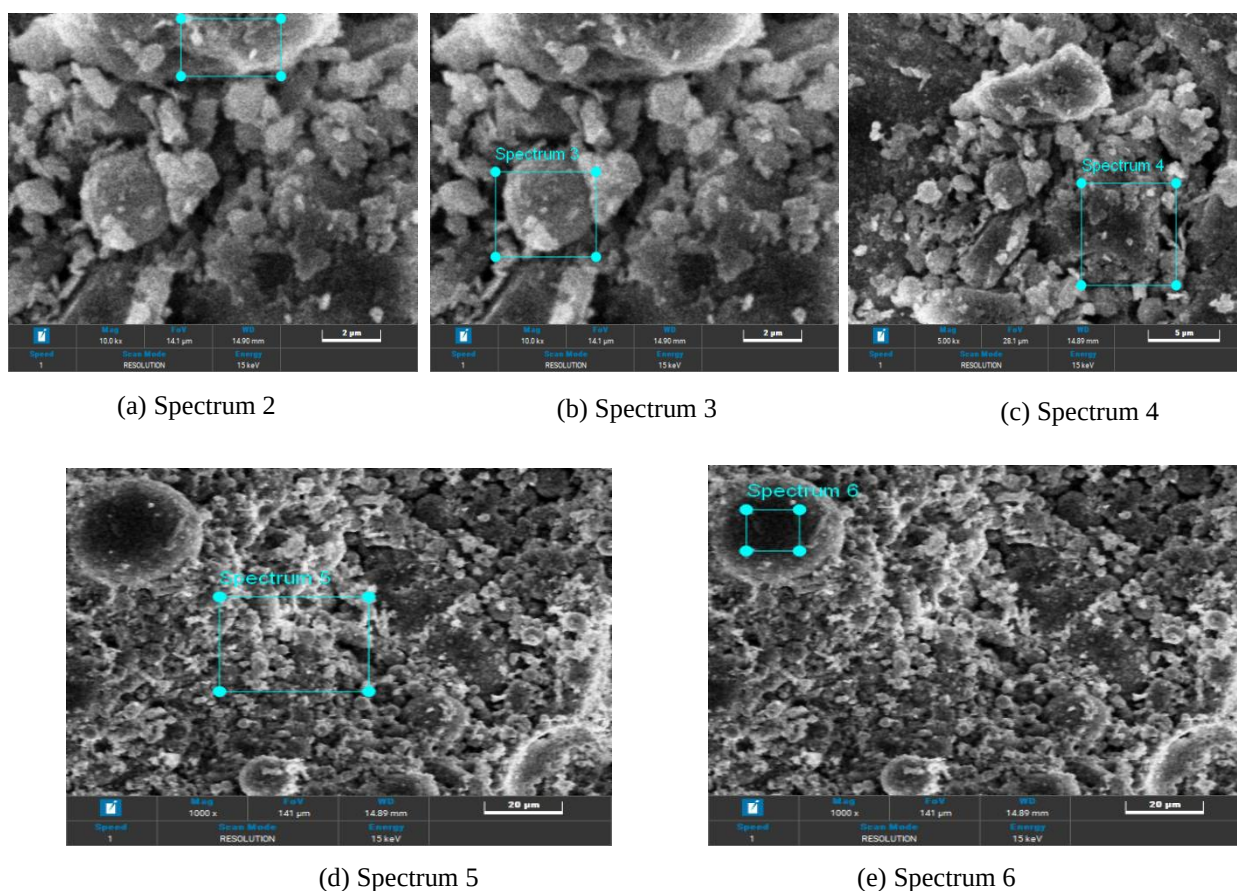


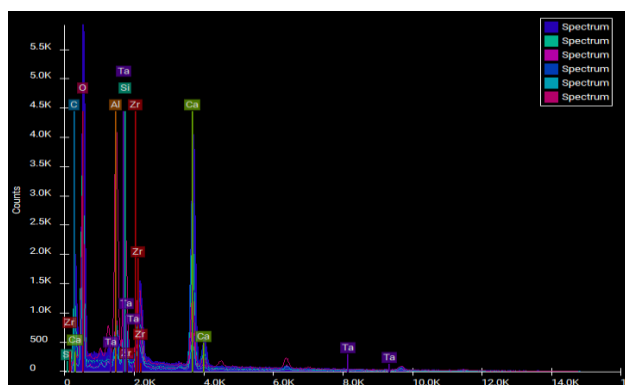
Fig. 4. Electron Microscope Images of the Composite Material at different Spectra.

Spectral analyses include scanned areas a little larger than the point analyses and yield area averaged compositional information. The wider range of bandwidth imparted in area spectra is indicative of the multiphase nature of a composite, typical of pozzolanic as well as cementitious systems and hence directly supports the XRD results (Fig. 5). Larger bandwidths with a greater number of overlapping peaks indicate the coexistence of diverse mineral phases such as Calcium-silicate-hydrate (C-S-H), Calcium-aluminosilicate-hydrate (C-A-S-H), Carbonate or oxide phases. The continued appearance of these peaks on all three spectra indicates the stability and the repeated formation of a phase in that sample.

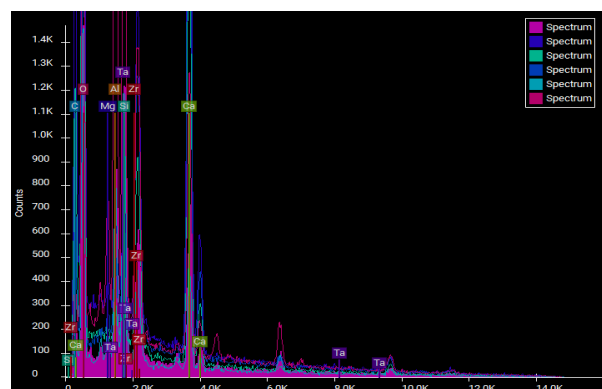
Chemical composition (atomic and weight percentage) analysis of elements in the material at

Spectrum 2-6 is reported in Table 2. It is interesting to note that the spectra 2-6 have nearly equal proportions of calcium, silicon, aluminum, and oxygen. This equilibrium is typical of cementitious systems in which calcium-rich phases share the same setting space as aluminosilicate frameworks. Differences in relative peak intensities across the spectra therefore indicate isolated regional disparities in particle composition rather than global heterogeneities and imply that the linear material remains morphologically homogeneous at the microscale.

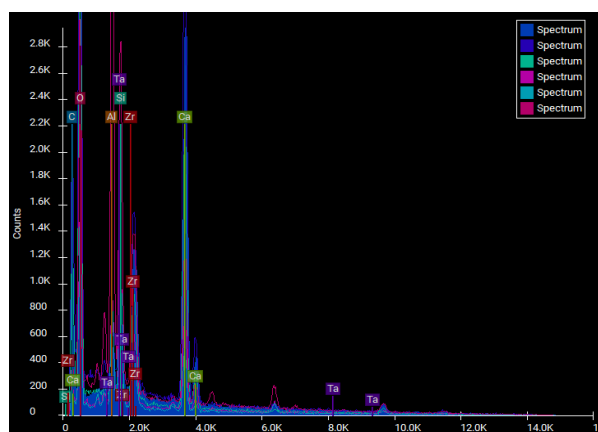
The higher oxygen content relative to heavier elements indicates the prevalence of oxide materials, which are less vulnerable to long-term sulphates and aggressive leachate.



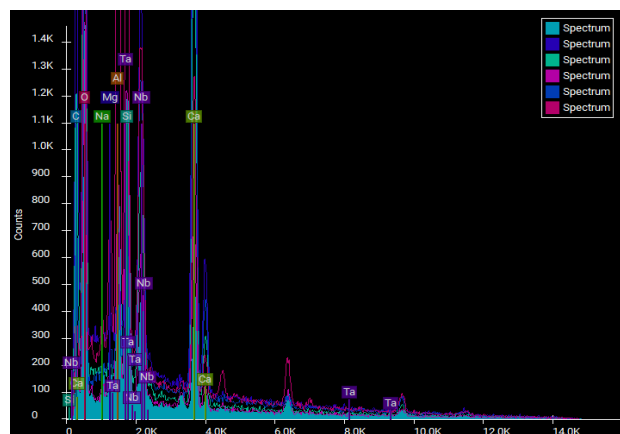
(a) Spectrum 2



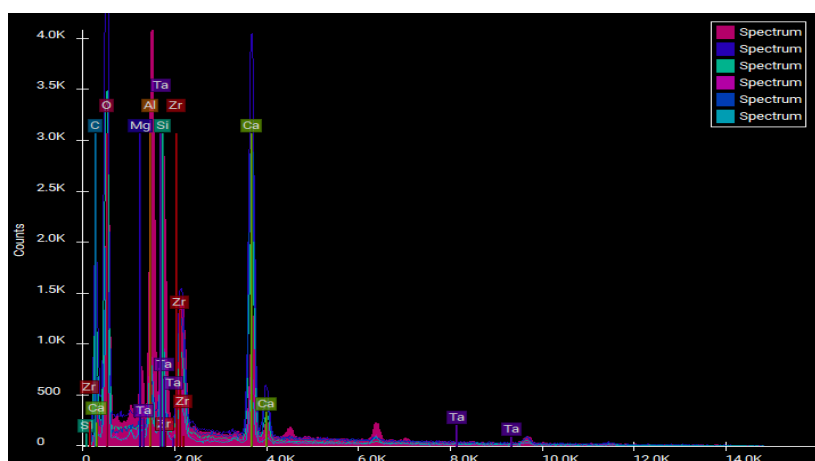
(b) Spectrum 3



(c) Spectrum 4



(d) Spectrum 5



(e) Spectrum 6

Fig. 5. Spectrum Bandwidth of the composite material at different spectra.

Table 2. Percentage of Atomic and Weight in terms of inherit elements of the material at different spectra.

Element	Spectrum 2		Spectrum 3		Spectrum 4		Spectrum 5		Spectrum 6	
	Atomic %	Weight %	Atomic %	Weight %	Atomic %	Weight %	Atomic %	Weight %	Atomic %	Weight %
Carbon	39.93%	27.06%	31.36%	18.71%	36.51%	20.28%	29.39%	17.10%	30.34%	17.16%
Oxygen	47.35%	42.75%	45.18%	35.91%	41.04%	30.36%	46.82%	36.28%	38.82%	29.24%
Sodium	-	-	-	-	-	-	1.07%	1.19%	-	-
Magnesium	-	-	0.76%	0.92%	-	-	1.02%	1.19%	2.09%	2.39%
Aluminum	0.81%	1.23%	5.58%	7.48%	1.43%	1.78%	5.83%	7.62%	12.31%	15.64%
Silicon	0.74%	1.17%	6.80%	9.49%	0.28%	0.36%	7.15%	9.73%	7.29%	9.64%
Calcium	10.40%	23.53%	8.37%	16.66%	18.41%	34.12%	6.07%	11.79%	6.62%	12.49%
Zirconium	0.70%	3.62%	1.50%	6.78%	1.56%	6.59%	1.89%	8.51%	1.90%	8.16%
Tantalum	0.06%	0.64%	0.45%	4.05%	0.78%	6.50%	0.75%	6.59%	0.62%	5.28%

Discussion

Mechanical and Physical Performance

The chosen FA: WC: CKD mix performed better than all other trials. The compressive strength of 1.64 MPa is higher than the range of compaction requirements (minimum) for liner materials, which suggests adequate mechanical stability to bear overburden pressure and operational stresses (Rowe, 2005).

A high fines content (71%) made the pore spaces smaller and the particle packing denser, which is crucial for obtaining low hydraulic conductivity. The specific gravity of 2.42 is contributed by FA and minerals, which help generating dense matrix liner.

Microstructural Characteristics from SEM Analysis

As magnification increased in the SEM images, a denser, and more compacted microstructure with smaller pores and better particle interlocking was

observed. At low magnifications (30× - 1000×) the material showed a fairly uniform matrix with few macro-voids. Further magnifications (5000×-10,000×) revealed compact particles and efficient subsidiary phases filling between grain boundaries.

This densification strongly supports superior mechanical performances, as reduced pore connectivity inhibits crack and fluid propagation. Analogous microstructural development was previously found in Calcium-silicate-hydrate (C-S-H), Calcium-aluminosilicate-hydrate (C-A-S-H), and Carbonate or oxide phases hydrating via pozzolanic or cementitious reactions. Fine WC particulates occupied intergranular voids, whereas products of reaction between FA and CKD acted to cement WC particles together. This tight microstructure is beneficial for reducing leachate migration and enhancing the service life of liners. (Scrivener et al., 2018).

Elemental Composition and Distribution (EDS Analysis)

The EDS spectrum bandwidths across all positions and spectra were representative of localized and area-averaged elemental distributions in the composite. The characteristic (peak) positions and intensities of oxygen, calcium, silicon and aluminum remain consistent across all spectra, implying chemical homogeneity and phase stability. Small differences in bandwidth indicate local compositional variance arising from calcium-rich hydration products and aluminosilicate phases. The wider regions in area spectra validate the material's multiphase behavior, distinct from pozzolanic and cementitious systems, indicating that SEM & XRD results are well established.

EDS point- and spectral analyses for all investigated spots confirm that these points indeed arise from carbon, oxygen, and calcium. The high oxygen content indicates oxide and silicate phases, while the high calcium content (~28 wt%) suggests the formation of a calcium-based binding material; such materials often improve both strength and durability. The high calcium content, mainly ascribed to CKD, is an important factor for promoting pozzolanic reactions with aluminosilicate phases of FA. (Neville, 2011).

Aluminium and silicon were recorded in reasonable amounts across many spectra, indicating that aluminosilicate and calcium-silicate-hydrate (C-S-H) type phases occurred in moderate abundance. These phases are also considered to be key contributors to mechanical strength and low permeability in synthetic materials (Taylor, 1997). Trace elements were present in small amounts and did not demonstrate any adverse effects, confirming that this composite is suitable for landfill liner applications due to its chemical compatibility.

Items with various concentrations of hafnium, zirconium, tantalum, and niobium showed significantly higher weight percentages, even though the atomic percent was relatively low. Their presence is important because it has been

demonstrated that they confer higher thermal stability, corrosion resistance, and resistance to chemical degradation (Lütjering and Williams, 2007). The detection of these features across multiple lines and spectra is strong evidence for reinforced abundances that are well mixed, rather than occurring as single explosive elements.

Mineralogical Insights from XRD

XRD study supported the SEM–EDS observation that crystalline calcium and silicate phases contributing to strength development and long-term stability were detected. The detected diffraction peaks are attributed to reaction products expected from pozzolanic activity and an increased matrix densification. These phases are responsible for the observed mechanical strength and microstructure densification. Coexistence of crystalline and amorphous phases indicates a stable composite material that can deliver performance in extended contact with leachate.

The coexistence of crystalline and amorphous phases indicates a combined or composite microstructure in which the crystalline compounds provide structural stiffness and the amorphous or semicrystalline structure promotes crack bridging and stress redistribution. This combined effect is particularly useful for applications that need good mechanical and environmental sustainability (Cullity and Stock, 2014).

Implications for Landfill Liner Performance

Together, the SEM–EDS and XRD results show a close relationship between the densifying microstructure, elemental composition, and mineralogical transformation. First, calcium- and silicate-containing phases decrease pore connectivity, and aluminosilicate bonding increases matrix cohesion. Other refractory trace elements such as zirconium and tantalum also improve corrosion, wear, and high-temperature resistance.

Taken together, these features suggest that the composite heterostructures should ensure a strong internal structure while maintaining strength and

stability in high-performance applications. The coupling of mechanical, micro-structural and mineralogical findings suggests that the FA–WC–CKD composite can be utilised effectively as a landfill liner material. Depleted pore connectivity, improved bonding, and chemically resistant phases all support long-term leachate retention, especially in landfills without engineered leachate collection systems.

SEM images corroborate these results by demonstrating densification and reduce pore connectivity, while EDS analysis shows low porosity and improved chemical resistance in samples containing calcium-rich/siliceous phases. XRD analysis further supported the crystalline compounds related to pozzolanic reactivity, leading to long-term performance durability. Microstructural investigation using SEM–EDS and XRD confirmed this conclusion, highlighting dense calcium- and silicate-rich matrices promote low pore connectivity and durability.

Conclusion

This work illustrates the potential of using locally available fly ash, white clay and cement kiln dust to build a low-cost and environmentally friendly composite landfill liner system. The FA:WC:CKD blend with an optimum mix proportion showed high compressive strength and a dense structure, achieving a preferable specific gravity that fulfilled liner performance demands.

This study comprehensively investigated the microstructural, elemental, and mineralogical properties of the material in question with SEM–EDS and XRD techniques. SEM observations showed an abundance of dense, compacted microstructure with less pore connectedness, while EDS examination confirmed calcium-, oxygen- and silicate-based species in the majority to densify and bond the matrix.

The XRD result also confirmed the generation of crystalline phases related to pozzolanic and cementitious reactions, which are primary factors for long-term durability and chemical stability. They found that trace elements, like zirconium, tantalum,

and niobium, have a positive effect on the corrosion resistance and thermal stability.

Overall, the combined microstructural and mineralogical evidence confirms that the developed composite material has been upgraded with improved strength, durability, and resistance to environmental attacks, justifying its applicability for long-term use as a potential liner system for leachate confinement in landfill applications, especially in resource-poor environments.

Author contribution

Md. Mehedi Emon: Conceptualization, methodology, investigation, experimental work, data curation, formal analysis, visualization, and original manuscript preparation. Aiman Al-Odaini: Provided research guidance, interpretation of results, and critical review of the manuscript. DatoIr Jamaludin Bin Non: Responsible for technical guidance, interpretation of findings, and critical review and revision of the manuscript. All authors contributed to the discussion of the results, reviewed the manuscript, and approved the final version for publication.

Conflict of interest

The authors declare no conflicts of interest regarding the publication of this article.

References

- Aziz HA, Rahman RA and Hung YT. Waste materials for landfill liner applications: A review. *Environ. Eng.Manag. J.* 2015;14(2): 343-356.
- Bouazza A, Ali MA, Gates WP and Rowe RK. New insight on geosynthetic clay liner hydration: The key role of subsoils mineralogy. *Geosynth. Int.*2017; 24(2): 139–150.
- Cullity BD and Stock SR. *Elements of X-ray diffraction* (3rd ed.). Pearson, 2014.
- Daniel DE and Koerner RM. *Waste Containment Facilities*. ASCE Press,2007.
- Edil TB, Benson CH and Bin-Shafique MS. Recycling of industrial by-products in

- geotechnical engineering. *J. Mate. Civil Eng.* 2012; 24(10): 1305-1313.
- Goldstein JI, Newbury DE, Michael JR, Ritchie NWM, Scott JHJ and Joy DC. *Scanning Electron Microscopy and X-ray Microana.* (4th ed.). Springer, 2018.
- Lütjering G and Williams JC. *Titanium* (2nd ed.). Springer, 2007.
- Neville AM. *Properties of Concrete* (5th ed.). Pearson Education, 2011.
- Rowe RK, Brachman RWI, Hosney M, Take WA and Arnepalli DN. Insight into hydraulic conductivity testing of geosynthetic clay liners (GCLs) exhumed after 5 and 7 years in a cover. *Can.Geotech. J.* 2017; 54(8): 1118–1138.
- Rowe RK. Long-term performance of contaminant barrier systems. *Geotech.* 2005;55: 631-678.
- Scrivener KL, John VM and Gartner EM. Eco-efficient cements: Potential economically viable solutions for a low-CO₂ cement-based materials industry. *Cem.Concr. Res.* 2018; 114: 2-26.
- Taylor HFW. *Cement chemistry* (2nd ed.). Thomas Telford, 1997.
- Yoon S, Jeong JH and Kim S. Engineering properties of coal ash mixed materials for landfill liners. *Waste Manag.* 2009;29(2): 722-730.