

Expansivity mitigation of black clay soil using agro-waste based inorganic polymer cement for flexible pavement subgrade

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Abstract

The use of cement for deficient black clay soil (BCS) improvement is a long time practice. But given the huge negative contribution of cement to carbon emission and climate change and its high demand by the construction industry that led to its scarcity and expensiveness, there is a need for an alternative binder to cement. The possible use of two biomass based inorganic polymer cements (IPC) was investigated herein. Rice husk ash (RHA) and sawdust ash (SDA) from biomass were used for this study. All experimental tests were carried out in accordance with British Standards. Soil samples were mixed with ash-IPC in steps of 0, 5, 10, 15 and 20% of the dry weight of the natural black clay soil. Results indicated significant improvement in all the geotechnical properties tested. The percentage passing the #200 sieve was reduced from 76.25% to 24.34% and 35.51% by RHA-IPC and SDA-IPC respectively at 20% treatment. Peak UCS values of 1123.56 and 954.28 kN/m² were respectively recorded for RHA-IPC and SDA-IPC treatments at 20% IPC content and at 28 days curing period. That represents an 813% increment in UCS value at 20% RHA-IPC content and 675% for SDA-IPC. The CBR value increased by 1500 and 1233% respectively for RHA-IPC and SDA-IPC treatments. The Scanning electron microscopy and energy dispersive X-Ray spectrometer EDS results indicated obvious improvement in the particle sizes in the microstructure of the treated BCS. The achieved improvements in all parameters tested are indications that the expansivity of the weak BCS has been reduced to acceptable levels and the effectiveness of using RHA-IPC and SDA-IPC for subgrade soils improvement.

Keywords: expansive clay, inorganic polymer cement, sawdust ash, rice husk ash, geopolymer cements, flexible pavements

Kulcsszavak: expanzív agyag, szervetlen polimer cement, fűrészpor hamu, rizshéj hamu, geopolimer cementek, rugalmas burkolatok

1. Introduction

These days, suitable lands for engineering construction works in urban and metropolitan areas are scarce and expensive. The need for land for the purpose of housing and urban road networking has drastically increased due to the increase in population and migration to urban areas. This makes inevitable the utilization and construction on weak subgrades that are geologically characterized by poor geotechnical properties with low strength characteristics and high compressibility behavior. Improvement of these weak subgrade soils has proved to make more economic sense than replacing them with foreign competent materials [1]. This case becomes an issue of major concern when it comes to road networking around urban areas due to the high volume of materials required and haulage constraints, discomfort to the commuting vehicles and expensiveness. In the past, cement was the most commonly used soil improvement-agent. Cement has been used successfully to improve the strength and consistency properties of weak subgrade soils in both cold and hot climates [2-3]. However, due to the huge negative contribution of cement to climate change and emission of greenhouse gasses and its high demand in the construction industry that led to its scarcity and expensiveness, there is an

emergency need for alternative binders to cement. A promising, efficient and eco friendly alternative to cement could possibly be agro-industrial waste ash inorganic polymer cements such as rice husk ash-inorganic polymer cement (RHA-IPC) and sawdust ash-inorganic polymer cement (SDA-IPC).

Black clay soils (BCS) are inorganic clays of high compressibility. BCS is very hard when dry but completely loses its strength when soaked in water. They are characterized by high shrinkage and swelling properties. According to Salahudeen and Akiije [4], the poor geotechnical properties of BCS have made construction on or with them an extreme challenge. Upon drying, cracks of varying widths and depths develop in black clays. Due to wetting and drying processes, heaving and shrinkage take place in the soil deposit. These vertical movements lead to failure of structures and flexible pavements built with or on them. These failures appear in the form of settlement, heavy depression, routing, cracking and unevenness. BCS are highly expansive with free swell index of over 50% and extremely weak when wet having a CBR value of less than 3% grossly leading to road network distortion as a result of seasonal variation in moisture condition [5-7]. In this study the improvement of BCS was undertaken using two agricultural waste materials: rice husk ash and sawdust ash with alkaline activators. Rice husk is a biomass

discharged from the milling of rice paddy. The total weight of RHA during the milling process of paddy is around 22% [8]. Koteswara-Rao et al. [9] discovered that RHA contains about 80-85% of silica by oxide composition. The chemical composition and reactivity of the amorphous or crystalline RHA depends largely on the burning duration, incineration temperature and grinding method of the ash.

The disposal of RHA in landfills constitutes serious environmental pollution. The incineration of rice husk results in a residual ash known as rice husk ash (RHA). Mboya et al. [10] observed that when rice husk is sufficiently burnt under a controlled incineration process, it becomes pozzolanic which is capable of replacing cement at an optimum amount of incorporation. Treatment of deficient soils with rice husk ash has been reported to improve the load bearing capacity of the soil to a good extent. Together with RHA, sawdust ash based inorganic polymer cement (IPC) was also investigated and compared in this research. Sawdust is biomass waste from the wood industry. The residue produced when the wood is sliced into timber of different sizes using saw-teeth is known as sawdust. The major chemical composition of sawdust is carbon (60.8%), oxygen (33.8%) and hydrogen (5.2%). Sawdust is highly combustible yet, the disposal of its residual ash is a very difficult challenge to the environmentalists [11]. Dumping of sawdust in the open environment causes aesthetic problems and air pollution which could lead to respiratory problems.

The BCS investigated in this study was treated with biomass based inorganic polymer cement (IPC) for the improvement of its geotechnical properties. Inorganic polymer cement (IPC), also known as geopolymers, is an alkali-activated aluminosilicate binder that is formed by reacting properly grinded ash that contains a sufficient amount of silica and alumina with a solution of alkali or alkali salts [12]. The mixture of silica and alumina materials results in crystalline-gels compounds forming a new matrix when hardened. Researchers [13] have reported the acceptability of IPC in improving the plasticity, shrinkage, bearing capacity, compressive strength, compressibility and durability characteristics of modified deficient soils. The geopolymerization process that determines the bonding strength soil properties improvement capability depends on the moisture/solid ratio, alkali concentration, heat energy, pH value, Al_2O_3/SiO_2 ratio, curing temperature and duration [14]. Two commonly used IPC of agro-industrial waste origin are RHA-IPC and SDA-IPC. The performance of these two polymer cements reported in the literature cannot be justifiably compared due to the varying properties and origins of the soils they were used to improve. This study investigated and compared the performance of the two biomass ashes

in geopolymerisation process with the same soil. The study aimed at using RHA-IPC and SDA-IPC as BCS improvement admixtures in flexible pavement subgrade applications.

2. Materials and experimental procedures

This experimental design includes stepped addition of IPC to the soil in concentration of 0, 5, 10, 15 and 20% of the dry weight of the soil. A maximum amount of 20% of IPC was considered due to economic concern in higher amounts.

2.1 Materials

Black clay soil

The black clay soil used for this study was obtained from a borrow pit as disturbed sample at a location within Latitude $10^{\circ}13'N$ and longitude $11^{\circ}23'E$. The location map is presented in Fig. 1. The oxide composition analysis of the BCS was carried out using X-ray fluorescence spectrometer as presented in Table 1.

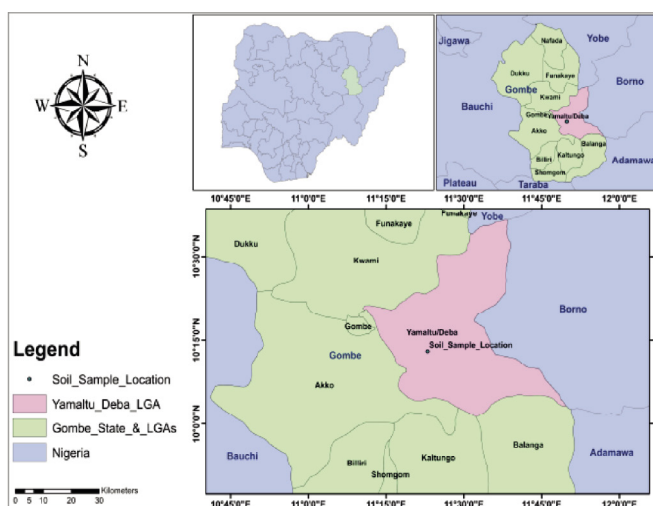


Fig. 1 Soil site location map
1. ábra: Talajtelephely térképe

Rice husk ash (RHA)

The rice husk used was locally obtained from a rice mill factory. RHA was produced by burning the dried rice husks in an open air. The burning process continued in an open air temperature for about a week. When the rice husk burnt to ash and cooled, the ash was sieved through sieve No. 200 (75 μm opening) to obtain the used component. The oxide composition analysis of RHA was carried out using X-ray fluorescence spectrometer as shown in Table 1. It was observed that the combined content of calcium oxide and silicon oxide for RHA and SDA are 67.94 and 69.51% respectively.

Oxide	SiO ₂	Al ₂ O ₃	SO ₃	K ₂ O	CaO	Fe ₂ O ₃	MgO	P ₂ O ₅	LOI *	Ag ₂ O	MnO	ZnO	PbO	TiO ₂	NaO	MnO ₂
RHA	64.2	6.4	0.91	4.68	3.74	6.42	4.8	3.6	4.65	-	0.43	0.2	0.06	0.64	-	0.35
SDA	65.3	6.09	3.12	9.1	4.21	3.63	3.39	-	12.8	-	0.69	2.52	9.1	0.4	1.00	-
BCS	49.4	15.1	-	2.25	3.58	14.2	-	-	11.1	2.17	0.23	-	-	2.09	-	-

*Loss on ignition

Table 1 Oxide composition of materials (concentration in %)
1. táblázat Anyagok oxidos összetétele

Property	Value	Property	Value
% passing #200 sieve	76.25	NBRI Classification	High swell potential
Liquid limit (%)	49	NMC (%)	22.8
Plastic limits (%)	25	MDD (Mg/m³)	1.65
Plasticity index (%)	24	OMC (%)	17
Linear Shrinkage (%)	22	CBR (%)	3
Free Swell (%)	82	UCS (kN/m²)	123.05
Specific gravity	2.41	Colour	Greyish black
ASHTO Classification	A-7-6 (28)	Dominant clay mineral	Montmorillonite
USCS	CL	-	-

Table 2 Properties of natural kaolin clay soil
2. táblázat Természetes kaolin agyag talaj tulajdonságai

Salahudeen et al. [7] reported that one of the compounds responsible for strength development in modified soils is calcium silicate hydrates. With the relatively high contents of these two oxides in the ashes used for this study, it is expected that IPC made of them will make significant improvement in the strength of the modified samples. However, the amount of loss on ignition content of an ash affects the strength yield of the sample since decreases with increase in loss on ignition content due to its increased content of volatile organic content.

Sawdust ash

The sawdust was obtained from a local registered sawmill. The same process performed on rice husk and RHA was also followed on sawdust and SDA. The X-ray fluorescence spectrometer results on sawdust ash are presented in Table 1.

Inorganic Polymer Cement

Two inorganic polymer cements were used in this study to modify black clay soil: rice husk ash-inorganic polymer cement (RHA-IPC) and sawdust ash-inorganic polymer cement (SDA-IPC). Preparation method of the ashes have been described above. The inorganic polymer cement (IPC) is a mixture of ash and alkaline activator. The alkaline activator used for this study is a combination of sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃). NaOH and Na₂SiO₃ mixture was prepared in ratio of 1:1 which together formed 44% of the IPC. Meaning, the ash content of the IPC is 56%. The NaOH and Na₂SiO₃ solution was prepared by mixing water with NaOH and Na₂SiO₃ in a metal container. The RHA-IPC was prepared separately and used independently from SDA-IPC.

2.2 Experimental Procedures

All experimental tests were carried out in accordance with British Standards. BS 1377(1990) for natural BCS and BS 1924 (1990) for the ash-IPC modified BCS. Soil samples were mixed with ash-IPC in steps of 0, 5, 10, 15 and 20% of the dry weight of the natural black clay soil. The experimental tests conducted include sieve analysis, compaction characteristics, consistency limits, unconfined compressive strength (UCS) tests and California bearing ratio (CBR). The moisture-density test was carried out using the British Standard light compactive effort. The strength tests performed are the California bearing ratio (CBR) and unconfined compressive strength (UCS) tests.

UCS samples were cured for 7, 14 and 28 days before the specimens were tested. The preparation and testing of CBR samples were carried out in accordance with the BS procedures and then based on the recommendation of the Nigerian General Specifications (1997) that required CBR specimens to be cured for six days in open air and then soaked for 24 hours before testing.

Scanning Electron Microscopy

The microstructure of the natural and ash-IPC modified BCS were studied after 28 days curing period. Two nanotechnological methods: Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray Spectrometer (EDS) were used to study the microstructure of the specimens using a SEM model “Tecan Vega SEM”. Some selected spots of the SEM were analyzed using the EDS. Surface roughness effect was prevented by polishing the specimens since the electron probe penetrates just to a shallow depth.

3. Results and analyses

3.1 Geotechnical Properties of the Untreated Black Clay Soil

The BCS used in this study was classified as A-7-6 soil according to AASHTO (1986) and CL using the ASTM (1992). The soil’s properties are summarized in Table 2. It is obvious from Table 2 that the soil will be unsuitable for road construction or any civil engineering application in its natural state. For any geotechnical applications, subgrade soil that has CBR value of 3%, plasticity index of 24%, free swell of 82% and having 76.25% of its portion passing #200 sieve has to be treated for property improvement before any application.

3.2 Expansivity mitigation by Particle Size and Consistency Improvement

The particle size distribution curves of the natural BCS and IPC treated soil samples are presented in Fig. 2. It is obvious that as the IPC content is increased, the curves shifted downward indicating a decrease in percentage fines content which is an appreciable improvement in the properties of the soil. The two IPC used (RHA-IPC and SDA-IPC) performed excellently at higher contents in improving the particle sizes of the soil. From the 76.25% passing the #200 sieve for the natural

BCS, 20% content RHA-IPC reduced it to 24.34% while it was reduced to 35.51% by SDA-IPC at 20% treatment. Soils having less than 50% of the portion passing the #200 sieve are classified as coarse by Unified Soil Classification System in ASTM (1992). In these results, there is an indication of cementation reaction between IPC and clay minerals of BCS which enhanced coagulation of the fine particles to form larger particles (Salahudeen et al, 2014). Expansivity of soils decreases with decrease in their fine content which has been reduced by 68.08% using the RHA-IPC and by 53.43% using the SDA-IPC.

(1997) that specified a maximum value of 12% plasticity index for materials to be used for road sub-base. It was observed that a lower amount of RHA-IPC is required for efficient improvement of BCS compared to SDA-IPC.

3.3 Moisture-Density Properties of IPC Treated BCS

The maximum dry density (MDD) and optimum moisture content (OMC) of a soil are respectively used to determine the amount of compaction densification and the required water content to achieve it both in the laboratory and on the field. It has a direct link with the strength potential of the soil. The variation of compaction characteristics of treated and untreated BCS is presented in Fig. 4. The MDD and OMC increased continuously with increased content of RHA-IPC and SDA-IPC. Soil stabilization scientists [16-17] are of the opinion that increase in MDD with chemical treatment could be as a result of the flocculation and agglomeration of clay particles due to exchange of ions at the clay surface. The increase in OMC may be due to additional water demands for the formation of $\text{Ca}(\text{OH})_2$ compound and its dissolution into Ca^{2+} and OH^- ions which is required to release more Ca^{2+} ions for cation exchange reaction for strength gains. It could also be due to increased demand for moisture to balance up with the higher content of IPC needed for its hydration reaction. The MDD increased from 1.65 Mg/m^3 for the untreated soil to a peak value of 1.82 Mg/m^3 when treated with 20% RHA-IPC. For SDA-IPC treatment, the increment was from 1.65 Mg/m^3 for the untreated soil to a peak value of 1.78 Mg/m^3 at 20% treatment. These observations in the outcome of MDD and OMC are appreciable improvements on the natural weak BCS.

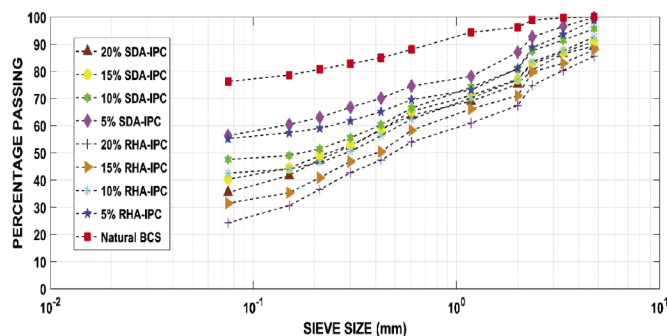


Fig. 2 Particle size distribution curves of natural and inorganic polymer cement modified black cotton soil

2. ábra Természetes és szervetlen polimercementtel módosított fekete pamuttalaj szemcseméret-eloszlási görbéi

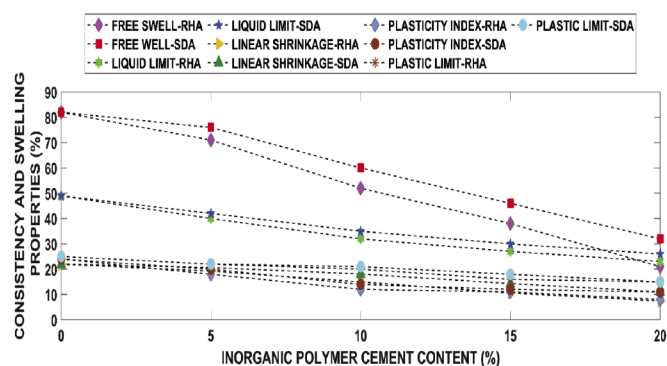


Fig. 3 Consistency limits and free swell of natural and inorganic polymer cement modified black cotton soil

3. ábra Természetes és szervetlen polimercementtel módosított fekete pamut talaj konzisztencia határai és szabad duzzadása

The consistency limits on the other hand, are important soil characterization and classification parameters. The lower the plasticity index of a soil, the lower is the clay content present in them and therefore the lower will be the expansivity potential of the soil. Clean sands are non-plastic because of the absence of clay content in them. The variation of Atterberg limits and free swell of treated and untreated BCS with IPC content are shown in Fig. 3. The treatment of the soil with both RHA and SDA IPC yielded a continuous decrease in the free swell and Atterberg limits. Suhail et al. [15] concluded that the decrease in free swell and plasticity of the soil admixed with chemical is as a result of the depressed double layer thickness due to introduced pozzolanic substances and cation exchange reaction by calcium, potassium and ferric ions. The plasticity index value of the natural BCS of 24% decreased to 12% at 10% RHA-IPC content and also to 12% at 15% SDA-IPC content. This result satisfied the recommendation of the Nigerian General Specifications

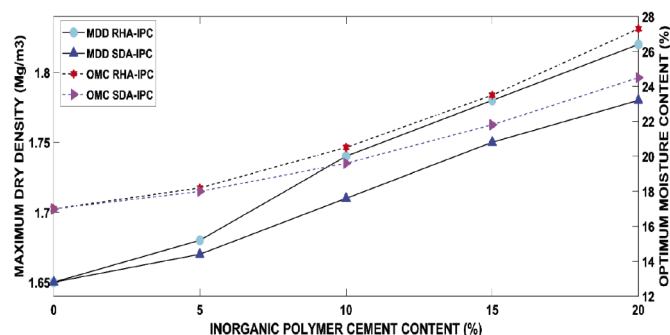


Fig. 4 Compaction characteristics of natural and inorganic polymer cement modified black cotton soil

4. ábra Természetes és szervetlen polimercementtel módosított fekete pamuttalaj tömörítési jellemzői

3.4 Strength Characteristics of IPC Treated BCS

Two strength tests for subgrade soils were considered in this study. They are the unconfined compressive strength (UCS) and California bearing ratio (CBR) tests. The UCS indicates the load bearing capacity of the soil under axial compression while the 6 days cured and 24 hours soaked CBR is used to determine the durability and resilience of the loaded subgrade soil in a harsh environment. The variation of UCS of BCS with IPC content for 7, 14 and 28 days curing periods are shown in Fig. 5. A general improvement was observed in the UCS values with both curing period and IPC content. Osinubi et al. [18] and Sadeeq and Salahudeen [19] opined that increase in UCS values could be as a

result of calcium aluminate hydrates and calcium silicate hydrates formations and improvements in the micro contents. When Ca^{2+} in IPC chemically reacted with the lower valence metallic ions in the BCS microstructure during ion exchange at the surface of clay particles which resulted in agglomeration of clay particles, the soil matrix gained more strength. Peak values of 1123.56 and 954.28 kN/m^2 were respectively recorded for RHA-IPC and SDA-IPC treatments at 20% IPC content and at 28 days curing period. That is, the RHA-IPC treatment caused an increment of 813% in UCS value at 20% IPC content while the SDA-IPC caused 675% increment also at 20% treatment after 28 days curing period. These improvement is significant to support the fact that the expansivity of the weak BCS has been reduced to acceptable levels.

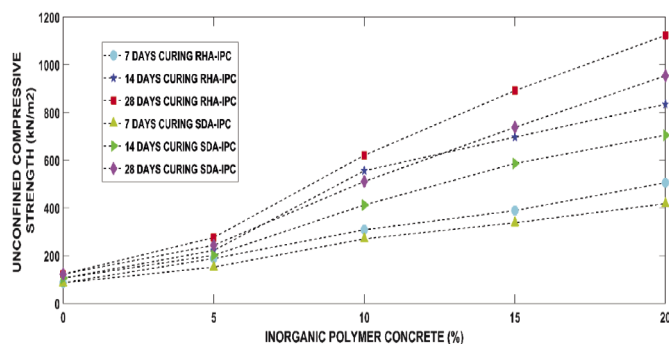


Fig. 5 UCS of natural and inorganic polymer cement modified black cotton soil
5. ábra Természetes és szervetlen polimer cementtel módosított fekete pamut talaj UCS

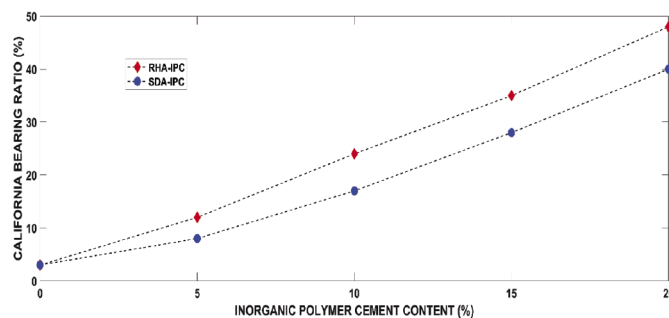


Fig. 6 CBR of natural and inorganic polymer cement modified black cotton soil
6. ábra Természetes és szervetlen polimer cementtel módosított fekete pamut talaj CBR

The variation of the 6 days cured and 24 hours soaked CBR values of the BCS with IPC content are presented in Fig. 6. The CBR values generally increased with increase in IPC content. This increase may be as a result of the abundant quantity of Ca needed for the formation of calcium aluminate hydrate and calcium silicate hydrate, the two major chemical compounds that bring about strength development in chemically treated soils. Peak CBR values of 48 and 40% were observed for the RHA-IPC and SDA-IPC treatments respectively at 20% IPC content from a value of 3% for the natural BCS. That represents 1500 and 1233% increments respectively for RHA-IPC and SDA-IPC treatments. It has been recommended [5] that CBR values of 20 – 30% for sub-base materials compacted at OMC is sufficient. The achieved UCS and CBR values are an indication of effectiveness of using biomass based inorganic polymer cements (IPC) for subgrade soils improvements with confirmation of rice husk ash (RHA) and sawdust ash (SDA) sources.

3.5 Microstructural Analyses of IPC Treated BCS

The results of scanning electron microscopy (SEM) together with the energy dispersive X-Ray spectrometer (EDS) analyses of 28 days cured samples of natural BCS, 10% IPC-RHA treated BCS and 10% IPC-SDA treated BCS are presented in Fig. 7, 8 and 9 respectively.

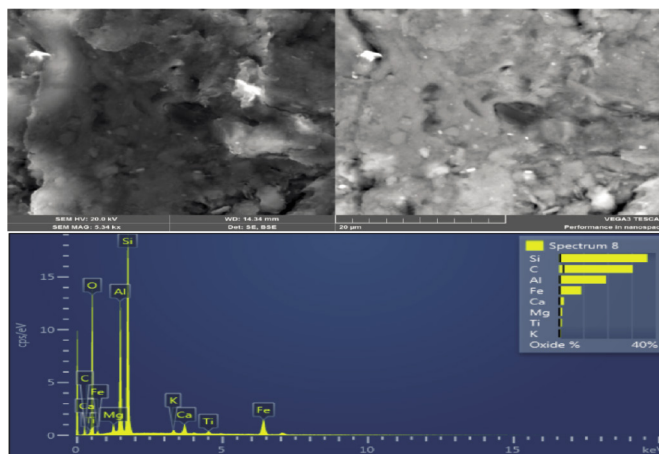


Fig. 7 SEM and EDS of natural BCS
7. ábra Természetes BCS SEM és EDS felvétele

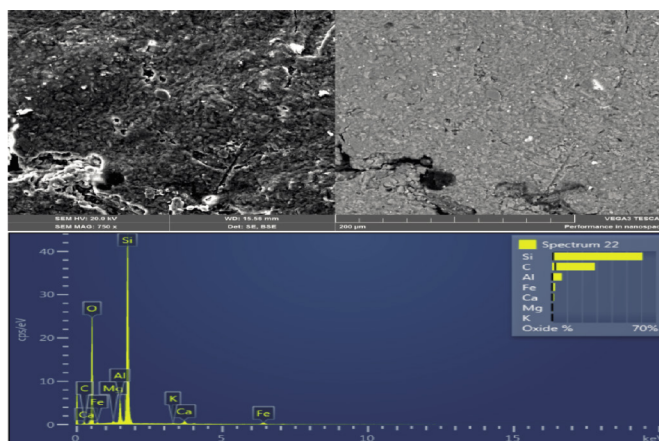


Fig. 8 SEM and EDS of 10% IPC-RHA treated BCS
8. ábra 10%-os IPC-RHA-val kezelt BCS SEM és EDS felvétele

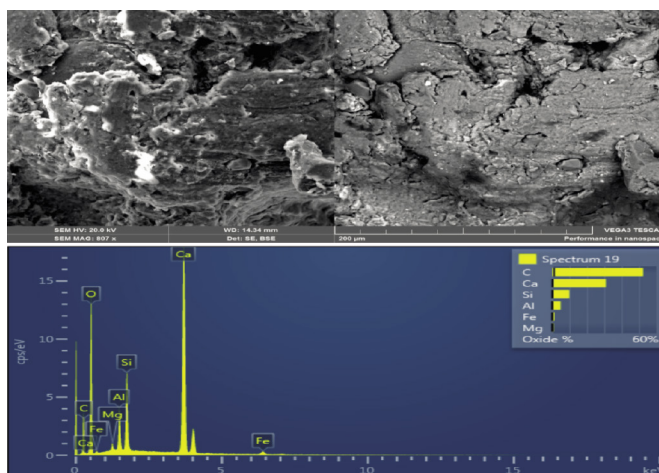


Fig. 9 SEM and EDS of 10% IPC-SDA treated BCS
9. ábra 10%-os IPC-SDA-val kezelt BCS SEM és EDS felvétele

The aluminosilicates mineral composition was observed in the EDS elemental analyses of the natural BCS. It consists majorly of Si, Al and Fe with some Ca content. The total oxide (%) as detected by the EDS for the natural soil is 40%. This value increased to 70% with 10% IPC-RHA content and 60% after IPC-SDA treatment. Reyes et al. [20] noted that the presence of elemental Carbon in the EDS is as a result of the carbon tape at the background of the sample holder attached to the machine. It was clear that the aggregate particles in the natural BCS SEM are smaller size distributions compared to those of IPC-RHA and IPC-SDA treated specimens. This could be due to the reactions of the higher valent cation, Ca^{2+} , which is also more active in the mixtures replaced the weakly bonded ions in the clay structure. Obviously, the higher strength values recorded at 28 days curing period could be as a result of the larger particle sizes formed in the BCS treated with 10% ash-IPC.

4. Conclusions

This study investigated the possible use of rice husk ash (RHA) and sawdust ash (SDA) admixed inorganic polymer cements (IPC) for improving black clay soils (BCS) in flexible pavement foundation application. The natural BCS was classified as A-7-6 and has CBR value of 3%, plasticity index of 24%, free swell of 82% and having 76.25% of its portion passing the number 200 sieve. This soil has geotechnical properties deficiency and was therefore improved to meet standard requirements. Following are conclusions drawn from this study results.

- Both RHA-IPC and SDA-IPC performed excellently in improving the particle sizes of the soil. From the 76.25% passing the #200 sieve for the natural BCS, 20% content RHA-IPC reduced it to 24.34% while it was reduced to 35.51% by SDA-IPC at 20% treatment. Likewise, the plasticity index value of the natural BCS of 24% decreased to 12% at 10% RHA-IPC content and to 12% at 15% SDA-IPC content. These are significant improvements in the expansivity properties of the soil.
- The MDD and OMC continuously increased RHA-IPC and SDA-IPC contents. The MDD increased from 1.65 Mg/m³ for the untreated soil to a peak value of 1.82 Mg/m³ when treated with 20% RHA-IPC. For SDA-IPC treatment, the increment was from 1.65 Mg/m³ for the untreated soil to a peak value of 1.78 Mg/m³ at 20% treatment.
- A general improvement was observed in the UCS and CBR values with IPC content. Peak UCS values of 1123.56 and 954.28 kN/m² were respectively recorded for RHA-IPC and SDA-IPC treatments at 20% IPC content and at 28 days curing period. That is, the RHA-IPC treatment caused an increment of 813% in UCS value at 20% IPC content while the SDA-IPC caused 675% increment. Peak CBR values of 48 and 40% were observed for the RHA-IPC and SDA-IPC treatments respectively at 20% IPC content from a value of 3% for the natural BCS. That represents 1500 and 1233% increments respectively for RHA-IPC and SDA-IPC treatments.

- The SEM and EDS results indicated improvement in the particle sizes in the microstructure of the treated BCS. These significant improvements support the fact that the expansivity of the weak BCS has been reduced to acceptable levels. The achieved improvements for all parameters tested are indication of effectiveness of using biomass based inorganic polymer cements (IPC) for subgrade soils improvements with confirmation of rice husk ash (RHA) and sawdust ash (SDA) sources.

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