



Remediation Efficiency of Microwave Remediated Crude Oil Contaminated Black Cotton Soil (BCS) as Road Base Material

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ABSTRACT

Remediation of crude oil contaminated black cotton (COCBC) soil was carried out to obtain the remediation efficiency using microwave heat. Several laboratory tests were conducted on the COCBC soil to assess its suitability as a road construction material. Specimens were compacted using the British Standard Light (BSL) and British Standard Heavy (BSH) compactive efforts. The expansive black cotton soil was classified as A-7-6 (13) or CH according to the American Association of State Highway and Transportation Officials (AASHTO) and Unified Soil Classification System (USCS), respectively. Soils in these groups are regarded as unsuitable for engineering purposes. The 7-day, 14-day, and 28-day UCS gave peak values of 392.07 kN/m² at 10 hours, 423.85 kN/m² at 8 hours and 731.3 kN/m² at 8 hours of remediation, all under the BSH compactive effort. These values do not meet the 1710 kN/m² requirements specified by TRRL (1977) for base material. Only the UCS value after 28 days meet the requirement of 687-1373 kN/m² for sub-base as specified by Ingels and Metcalf (1972). Peak C.B.R values of 3.57% at 10 hours and 10.89% at 8 hours of remediation were obtained for BSL and BSH compactive efforts respectively. The values do not meet the specification for base or sub-base materials as outlined by the Nigerian General Specifications (1997) but met that of sub grade. Finally, the remediation efficiency of crude oil contaminated black cotton soil using microwave heat can be achieved after 8 hours of heating with remediation efficiency of 91%.

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INTRODUCTION

Crude oil is a vital natural resource that comes in a variety of forms and is the main source of energy (after refinement) for various vehicles, power plants, and other devices. As a result, it is a major driver of development in many nations around the world today, improving our quality of life and raising our standard of living. Many parts of the globe, including the United States, Russia, Romania, Iran, Mexico, Saudi Arabia, Iraq, Kuwait, Libya, and Nigeria, are home to it naturally. Each year, the petroleum industry produces billions of tonnes of natural gas, crude oil, and derivatives (Das and Chandran, 2011). Afterwards, all of these materials undergo additional processing to produce refined products

including lubricants, petrol, diesel and petrol (Ndimele *et al*, 2018). Global oil demand for liquid fuels is expected to increase by 1.1 million barrels per day in 2024 and 1.6 million barrels per day in 2025, according to estimates from the U.S. Energy Information Administration (EIA). According to the report, the global consumption of petroleum and other liquid fuels is expected to increase to 102.94 million barrels per day in 2024 and 104.55 million barrels per day in 2025. It is estimated that 101.80 million barrels of oil will be consumed daily in 2023.

Crude oil consists of nitrogen, sulphur, oxygen, and volatile liquid hydrocarbons (compounds mostly composed of hydrogen and oxygen). Many complex molecular structures are

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produced by those components, some of which are difficult to identify. Paraffins, naphthenes, and aromatics are the three types of hydrocarbon molecules that are most often found in crude oil. The most common hydrocarbon in crude oil is paraffin; certain liquid paraffins are the main ingredients in petrol (petrol) and are thus highly valued.

Pollution from petroleum hydrocarbons is becoming a major environmental issue on a worldwide scale. According to Das *et al.*, (2011), industrial discharge, storage tank breaches, and other events result in its release into the environment. According to Pinedo *et al.*, (2013), soil contaminated by petroleum is considered a hazardous waste that pollutes the environment both locally and widely. Crude oil allows carbon dioxide generated by soil organisms to be retained and forms a layer on the soil's surface. By binding the soil particles together, it also reduces the porosity of the soil (Ezeji *et al.*, 2007). The quantity and quality of dirt that spilled determine how much is lost.

The United States Environmental Protection Agency has declared polycyclic aromatic hydrocarbons (PAH) found in crude oil to be mutagenic and carcinogenic (Siles and Margesin, 2018). A prolonged contact time of stable PAH with soil induces a phenomenon known as soil ageing, which makes the soil resistant to any treatment (Garcia *et al.*, 2017), it further makes the soil resistant to treatment and limiting its use for engineering construction activities. Contaminant leakage from the soil to the groundwater can harm vegetation, human health, and the biological environment (Marinescu *et al.*, 2010). Akinwunmi (2014) discovered that contaminated soil reduced its permeability due to the entrapment of the crude oil within the soil's pore spaces. He further advice that such soil should not be used as a construction material if it is not remediated and possibly stabilized.

Black cotton soils (BCS) are major problematic soils in tropical regions especially in Africa and India. These materials are of low quality by temperate zone standards and are challenging to use in road and air field construction due to their high cost, primarily resulting from a significant

percentage of expansive clay minerals, i.e montmorillonite (Gidigas and Appeagye, 2013). Expansive soils are unreliable due to high volume and moisture sensitivity. The well-known type of expansive soil is black cotton soil (Sathyapriya *et al.*, 2023). These soils swell when they come into contact with water and contract as they dry.

The soil deposit are usually extensive, making avoidance impossible during engineering construction projects. Many roads and foundation of light buildings have been reportedly shown distress due to the seasonal volume changes (i.e swell and shrinkage) of these soils (Chen, 2008). These soils reportedly caused billions in damages and repairs yearly to earth structures and facilities. Various effective methods exists for remediating crude oil-contaminated soils which are efficient and dependable. Some of the methods are quick, nature friendly and cost effective. They include; chemical, physical, biological and thermal methods. However, the thermal remediation technique has many variations, including incineration, thermal desorption, smouldering, microwave (radio frequency) heating, steam enhanced extraction, electrical resistance heating, vitrification, and pyrolysis. We shall be making use of microwave heating method.

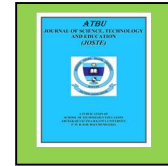
The thermal method involves thermal stripping, low temperature thermal desorption, or soil roasting contaminated soil to very low temperatures (200 to 1000 °F) (Ndimele *et al.*, 2018). Depending on the soil's organic compounds and the temperature at which the thermal stripping occurs, organic contaminants may be completely or partially decomposed. This method can remove about 90% of the contaminants, but it is very expensive and not environmentally friendly (Ndimele *et al.*, 2018). With its many benefits, including simultaneous and rapid selective heating (Zhang *et al.*, 2017), microwave heating (MWH) is regarded as a promising thermal remediation technology for contaminated soil. The microwave's mechanism involves subsurface penetration and heating of a high dielectric substance, allowing for low temperature treatment (Luo *et al.*, 2019). The aim of this research work is to evaluate the removal efficiency of crude oil contaminated BC soil using

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microwave heating technique and the soils suitability as a road construction material.

MATERIALS AND METHODS

Materials:

Black Cotton Soil:

The soil samples used for this study were collected by disturbed sampling at Biliri Local Government Area, along the Biliri Tangele-Ayabu road, which is 545km from Gombe state, at Latitude 10° 13'N, Longitude 11° 23'E. The topsoil was removed to a depth of 0.5m before taking the soil samples, which were then sealed in plastic bags and placed in sacks. The soil samples were then allowed to air-dry before being ground into particles that passed through BS No. 4 sieve.

Water: Tap water was collected from Department of Civil Engineering Soil Laboratory in Nigeria Defense Academy.

Crude Oil: To proceed with the necessary tests, the crude oil utilized in this study was delivered to the Civil Engineering Laboratory at the Nigerian Defence Academy Afaka from an oil mining location in Onne LGA of Rivers State, which is located in the Niger Delta.

Methods:

Index Properties:

Laboratory tests were carried out to determine the index properties of the soil mixtures by the British Standards BS 1377 (1990) and BS 1924 (1990) respectively. Some of the experiments conducted to obtain the desired results include, specific gravity, sieve analysis, Atterberg limit tests, compaction test, CBR test, unconfined compressive strength test, and durability analysis. Table 1 shows the summary of the index properties of the soil samples.

Table 1: Geotechnical properties of the natural and COCBC soil

Engineering Properties	Remediation Time (Hrs)					
	0	2	4	8	10	
Liquid limit, %	57.34	41.4	43.65	37.13	27	
Plastic limit, %	31.28	28.91	25.85	13.42	11.56	
Plasticity index, %	26.96	12.49	17.8	23.71	16.04	
Specific gravity	2.34	2.38	2.4	2.43	2.4	
Percentage passing BS No. 200 sieve	86.5	92.3	90.7	89.25	91.5	
AASHTO Classification	A-7-6	A-7-6	A-7-6	A-7-6	A-7-6	
USCS classification	CH	CH	CH	CH	CH	
MDD Mg/m ³	BSL	1.58	1.59	1.6	1.6	1.59
	BSH	1.61	1.62	1.63	1.65	1.63
OMC %	BSL	20.15	20.11	20.2	20.03	20.1
	BSH	15.51	15.42	15.02	14.85	15.24
CBR Soaked %	BSL	2.29	3.86	5.79	3.35	3.57
	BSH	3.4	8.37	5.29	10.89	5.83
Colour		Greyish black				
Dominant clay mineral		Montmorillonite				

Soil Contamination Process

Before the contamination, the BC soil was first of all, pulverized after subjecting it to airdrying process to completely remove the moisture present in the soil. This is to ensure that the crude oil is properly mixed with the soil sample.

Thereafter, the same percentage of crude oil and water, 6% and 11% respectively, by weight of the soil, were added to the various samples before mixing was done and allowed to cure for 14 days, and then, subjecting it to the microwave for the remediation process.

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Compaction

All compaction related to moisture-density relationships, CBR and UCS was conducted using energies derived from the British Standard Light (BSL) and British Standard Heavy (BSH). The BSL compaction was conducted using energy generated by a rammer of 2.5 kg falling from a height of 30 cm in a 1000 cm³ mould. The soil was compacted in three layers, each receiving 27 blows. The soaked CBR tests were conducted according to the Nigerian General Specifications (1997) which require that specimens be cured dry for 6 days, and then soaked for 24 hours before testing. The CBR compaction utilized the same rammer weight and drop height, with each layer receiving 62 blows in a 2360 cm³ mould.

The BSH compaction, was conducted using energy generated by a rammer with a mass of 4.5 kg falling from a height of 450 mm in a 1000 cm³ mould. The soil was compacted in five layers, each receiving 27 blows. The soaked CBR tests were conducted according to the Nigerian General Specifications (1997) which requires that specimens be cured for 6 days, and then soaked for 24 hours before testing. The CBR compaction involved the use of the same rammer weight and drop height with each layer receiving 62 blows in a 2360 cm³ mould.

RESULT AND DISCUSSION

Index Properties:

The soil is greyish black in colour (from wet to dry states), with a liquid limit of 57.34%, plastic limit of 31.28%, and plasticity index of 26.06. Butcher and Sallie (1984) and Osinubi and Medubi (1997), classified the soil as belonging to the CH group in the Unified Soil Classification System (ASTM, 1992) or A-7-6(13) group in the

American Association of State Highway and Transportation Officials soil classification system (AASHTO, 1986).

Soil Remediation Process:

The sample contaminant concentration level was found to be 42,200 mg/kg. This value exceeds the permissible limits for soils and thus needed to be remediated before its engineering usage (Egedeuzu and Nnorom, 2013; Meshari, 2021). The principle of microwave heating is based on the conversion of microwave energy adsorbed by the substances being irradiated into heat. This process depends on the dielectric constant and the dielectric loss factor of the materials (Falciglia et al, 2016; Zhou et al, 2016). The process of remediation was carried out in batches of samples with differing timing in hours, since the concentration level was the same for all samples.

The time variations were 2hrs, 4hrs, 8hrs and 10hrs respectively as seen in table 2. The respective samples were subjected to the microwave heating at a uniform power of 800 watts (0.8Kw). When each sample was transferred into the microwave, the power turned on and the time was set to count down to zero. Thereafter, the sample is removed and then further necessary tests including the removal efficiency were conducted on the various samples.

All the samples weighed the same (5,000mg) before the remediation, but differ in weight after the process, which is indicative of the remediation effects. After the remediation, there was a steady decrease in the concentration of samples from 2hrs up until the 8hrs, before a sharp increase at 10hrs.

Table 2: TPH contamination level for BC soil sample

S/N	sample ID	WT before extraction (mg)	WT after extraction (mg)	TPH content (mg/kg)
1.	COCS	5,000	4,789	42,200
2.	2HRS	5,000	4,829	34,200
3.	4HRS	5,000	4,866	26,800
4.	8HRS	5,000	4,980	4,000
5.	10HRS	5,000	4,915	17,000

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Total Petroleum Removal Efficiency:

The effectiveness of contaminant removal is affected by soil particle sizes (Khangee *et al*, 2020). Furthermore, he indicated that residual total petroleum hydrocarbon concentration decreased with increased

irradiation time, with the removal rate varying based on soil temperature changes. Table 3 and Fig.1. below shows a significant removal with an increase in time, from 2 hours to 8 hours, or 19% to 91% respectively.

Table 3: TPH Removal efficiency in percentage for soil sample

S/N	Sample ID	Removal efficiency (%)
1.	COCBCS	0
2.	2HRS	19
3.	4HRS	36
4.	8HRS	91
5.	10HRS	60

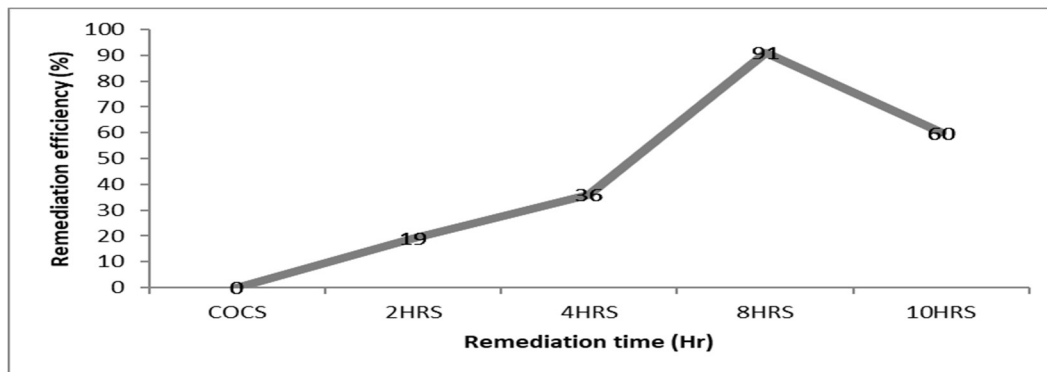


Fig.1: Removal efficiency of crude oil contaminated black cotton soil

Maximum Dry Density

The MDD for BSL and BSH compacting efforts aligns with the trend of increasing MDD (see Fig. 2). This increase in MDD is with respect to increased duration of heat applied is in

consistent with the work carried out by Lekan *et al*, 2024. Heat is said to reduce the cation exchange in clayey soi and increases its stiffness and density as reported by Geng *et al*, 2018).

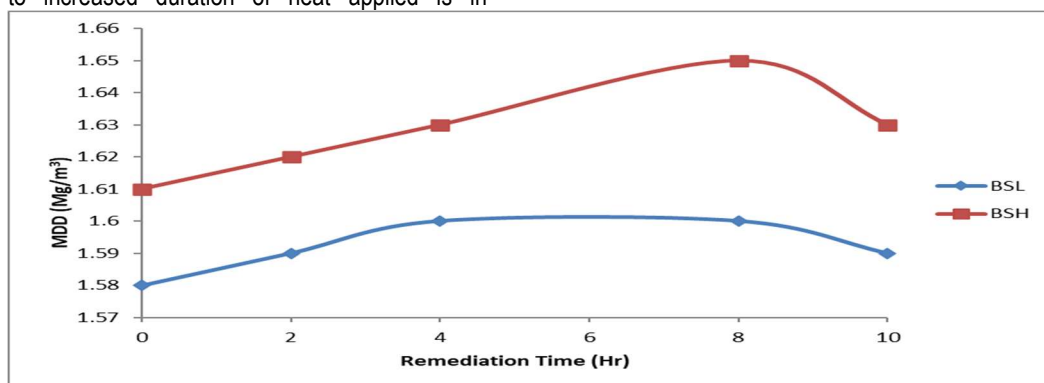


Fig. 2: variation of the Maximum Dry Density for the unremediated and remediated BC soil at BSL and BSH compactive efforts.

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Optimum Moisture Content

The compacting efforts at BSL and BSH for OMC showed a decreasing trend (see Fig. 3), likely due to self-desiccation and all the water were consumed, resulting in low hydration of the soils surface. This is due to the lengthy

remediation process which required additional water to lubricate the entire matrix, for increased surface area. This trend of decreasing OMC aligns with the submission by Geng *et al*, 2018 and Lekan *et al*, 2024.

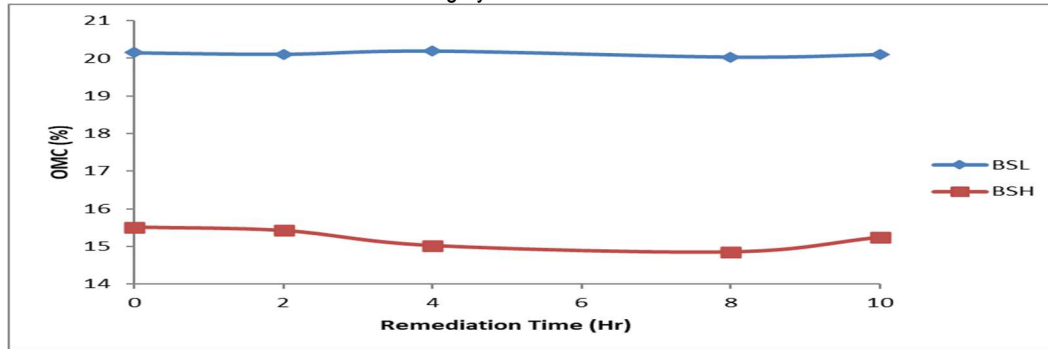


Fig 3: Variation of the Optimum Moisture Content of the unremediated and remediated BC soil

Strength Characteristics

Unconfined Compressive Strength

The variation of unconfined compressive strength (UCS) of the Black Cotton soil at different remediation time for 7-, 14-, and 28 days curing periods are shown in Fig. 4. In general, the UCS of the contaminated BC soil at the BSL and BSH compacting efforts increased for all curing periods but decreased after the 8 hours of remediation during the 14-day and 28-day curing periods. The 7 days UCS of the unremediated BC soil improved from a value of 281.84 kN/m² and 340.59kN/m² to 311.79kN/m² and 392.07kN/m² after the 10hour remediation for both BSL and BSH compactive efforts. These

values falls short of the 1710 kN/m² and 678-1373kN/m² specified by TRRL (1977) for base material and for sub-base specified by Ingels and Metcalf (1972) respectively. Similarly, the peak values for the 14 days curing period at BSL and BSH compactive efforts were 332.58kN/m² and 423.85kN/m², and the 28 days curing period at BSL and BSH compactive efforts were 448kN/m² and 731.3 kN/m² all after the 8 hours of thermal remediation. From the above result, it showed that only the BSH compactive effort can be used as sub-base material as specified by Ingels and Metcalf (1972).

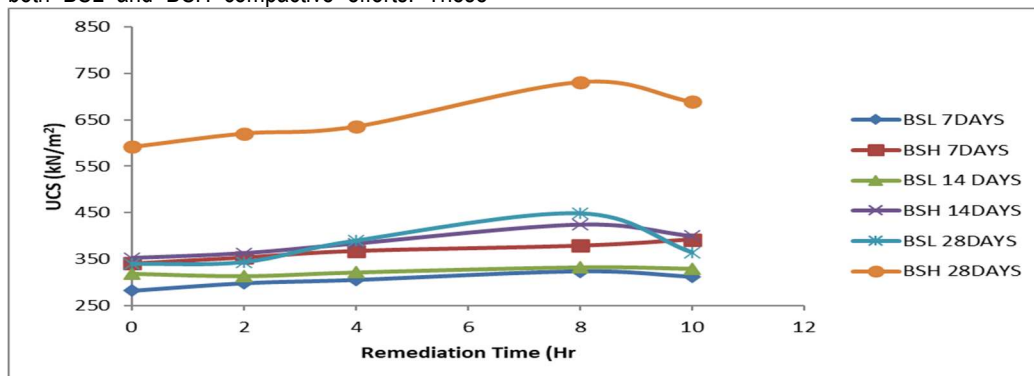


Fig. 4: Variations of the Unconfined Compressive Strength (7, 14 and 28 days curing periods) for the unremediated and remediated BC soil.

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California Bearing Ratio

The variation of California Bearing Ratio (CBR) of the un-remediated and remediated BC soil is shown in Fig. 5. Generally, the CBR increased with longer duration of remediation for both BSL and BSH compactive efforts. The reason for the increase is due to longer duration of heat applied. Although, the BSH compactive effort has a better CBR value than the BSL compactive

effort, it still failed to meet the minimum CBR value of 30 % specified by (BS 1990) for materials suitable for use as base course. Furthermore, none of the energy levels produced satisfactory values for base, sub-base material as specified by the Nigerian General Specifications (1997) but meet the requirement for sub grade specified by Nigerian General Specifications (1997).

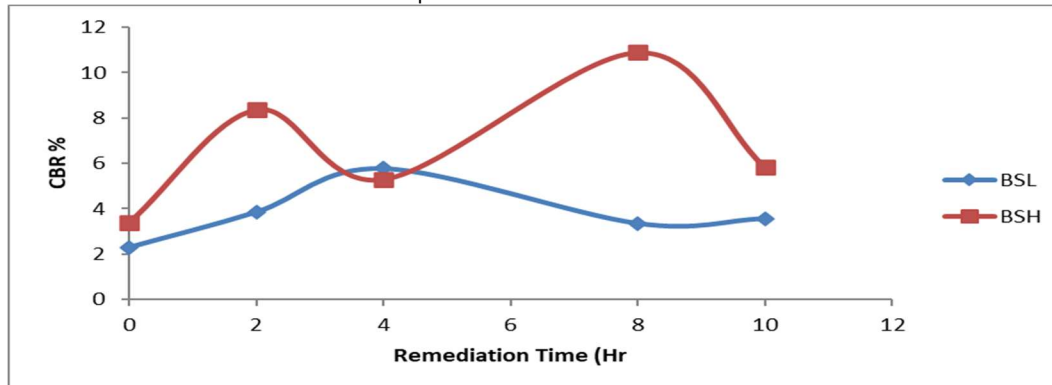


Fig. 5: Variations of the California Bearing Ratio (Soaked) for the unremediated and remediated BC soil.

CONCLUSIONS

The un-remediated and remediated COCBC soil from Biliri Tangele-Ayabu road was classified as A-7-6 in the AASHTO system and CH in the Unified Soil Classification System (USCS), respectively. Soils under these categories are of poor engineering characteristics.

The 7, 14, and 28 days UCS gave peak values of 392.07kN/m² at 10 hours, 423.85kN/m² at 8 hours and 731.3kN/m² at 8 hours of remediation, all under the BSH compactive effort. These values fall short of the 1710kN/m² specified by TRRL (1977) for base material. But only the 28 days UCS value meet the requirement of 687-1373kN/m² for sub-base as specified by Ingels and Metcalf (1972).

Peak C.B.R values of 3.57% at 10 hours and 10.89% at 8 hours of remediation were obtained for BSL and BSH compactive efforts respectively. These values do not meet the specifications for base or sub-base materials as outlined by the Nigerian General Specifications (1997) but the 10.89% recorded for BSH met the requirement for sub grade. Finally, the remediation efficiency of crude oil contaminated

black cotton soil using microwave heat can be achieved after 8 hours of heating with a 91% remediation efficiency. Furthermore, treatment of COCBC soil using microwave heat alone failed to yield the desired result for base materials. Therefore, soil stabilization following remediation is essential for road use.

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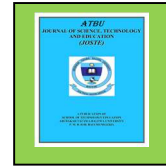
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