Vol. 10(3), pp. 60-70, July-December 2019

DOI: 10.5897/JCECT2019.0520 Articles Number: 611C48662510

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Journal of Civil Engineering and Construction Technology

Full Length Research Paper

Variability of visible and near-infrared (vis-NIR) diffuse spectral reflectance of cement-based solidified/ stabilized pre-treated oil-based drill cuttings

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Received 2 September, 19; Accepted 28 November, 2019

This study investigated the effects of solidification/stabilization (S/S) variables on visible and nearinfrared (vis-NIR) spectral reflectance of solidified/stabilized low-temperature thermally desorbed (pretreated) oil-based drill cuttings. Partial least squares (PLS) regression models were also developed to rapidly estimate the levels of total petroleum hydrocarbon (TPH), Cu, and Ba present in the solidified/stabilized matrix. Composite samples of the drill cuttings were collected from a drill cutting Treatment Company in the Niger Delta region (5.317°N and 6.467°E), Nigeria and subjected to cementbased S/S. Reference TPH analysis was done by gas chromatography mass spectrometry and heavy metals by flame atomic absorption spectrometry. Sample optical measurement was carried out with a portable fibre-optic LabSpec2500® vis-NIR spectrophotometer (350-2,500 nm) (PANalytical, Boulder, CO, USA). Results show that spectral absorption minima observed around 1411, 1759, 1950, and 2204 nm in the NIR band are linked to relics of hydrocarbon-based oil, oxides of metallic ions, sulphate, and water (but not necessarily in that order). Spectral reflectance increased with drill cuttings addition, days of curing, and water-to-cement ratio. PLS model predictions were almost good for Cu (validation residual prediction deviation [RPD] = 1.97) and excellent for both TPH and Ba (RPDs = 2.41 and 3.45, respectively). Hence, vis-NIR diffuse reflectance spectroscopy is a potential means for semiquantitative measurement of the levels of TPH and heavy metals present in cement-based solidified/stabilized pre-treated oil-based drill cuttings.

Key words: Characterization, hazardous waste management, near-infrared spectroscopy, solidification/stabilization, hydrocarbons, heavy metals

INTRODUCTION

It is widely known that huge quantities of petroleum wastes are usually generated during crude oil drilling

activities. These wastes are a complex mixture of both the solid and liquid components of the waste stream. The

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solid components are largely made up of drill cuttings, which comprise mixtures of soil and rock fragments; while crude oil, produced water, spent drilling mud, and mud additives make up the liquid components (Lyons, 1996).

It has also been widely acknowledged that drill cuttings are a hazardous waste; and this has been attributed to its chemistry. The chemistry of drill cuttings is largely influenced by the type of drilling mud used during drilling. As a result of the chemical formulation of the drilling mud, disposal of drill cuttings on land or sea has been reported to have impacted negatively on the environment (Okparanma et al., 2018).

At the moment, there are various secondary treatment options available for drill cuttings as reported in the literature (IOGP, 2016; Okparanma et al., 2018). As is usually the case in waste management, these treatment technologies may either be applied exclusively or combined to achieve the desired objective. For instance, thermal desorption followed by solidification/stabilization (S/S) of drill cuttings has been applied to facilitate the reuse of drill cuttings as partial replacement for cement or sand in concrete (International Association of Oil and Gas Producers (IOGP), 2016).

Thermal desorption is the indirect heating of drill cuttings to vapourize volatile and semi-volatile compounds without necessarily incinerating the drill cuttings (Okparanma et al., 2018). The aim of thermal desorption is to recover the solid components of drill cuttings for various types of application, especially in the construction industry (IOGP, 2016) and agriculture (Kogbara et al., 2017). Cement-based S/S utilizes the chemical reaction between the binding agents and drill cuttings to prevent the leaching of remnants of hydrocarbons, heavy metals, and water-soluble salts in the pre-treated drill cuttings (Drilling Waste Management Information Systems (DWMIS), 2000).

Before the solidified/stabilized matrix is put to the intended use, it is important that it is properly screened against environmental guidelines to be sure it is in compliance with the guideline values for the protection of both the present and future environmental health as recommended in the literature (IOGP, 2016). To do this, the solidified/stabilized matrix is first of all characterized for contaminants of potential ecological concern. The observed concentrations of target contaminants are then compared with environmental guidelines to check if they are below or above the guideline values. This ensures that the necessary actions (if need be) are taken to prevent problems that may arise in the future when the solidified/stabilized matrix is subjected to the intended use. However, the conundrum has been with the conventional techniques of characterizing the solidified/ which involve lengthy stabilized matrix, sample preparation protocols including sample crushing, acid digestion of crushed sample, solvent extraction, and chemical analysis. Sample preparation is time consuming and sample crushing is tedious. Both acid digestion and solvent extraction have associated health risks because toxic chemicals are often used in both processes. Instrumental analysis is a huge financial burden due to high analytical cost. These challenges are now driving research towards alternative analytical approaches that are cheap and fast such as visible (vis) and near-infrared (NIR) diffuse reflectance spectroscopy. Vis-NIR diffuse reflectance spectroscopy is also non-invasive, portable, and ecologically friendly (Okparanma and Mouazen, 2013a). Rapid characterization would quickly provide the necessary information to expedite decision making on the reuse of the solidified/stabilized matrix for construction qualitative, semi-quantitative, purposes. For quantitative applications of vis-NIR diffuse reflectance spectroscopy in soil analysis, chemometric techniques such as partial least-squares (PLS) regression analysis are often used. This is because vis-NIR diffuse reflectance spectroscopy depends on soil physical properties including particle size, colour, and moisture content, etc., which are highly variable (Pasquini, 2003). Vis-NIR diffuse reflectance spectroscopy has been widely used for soil characterization (Adamchuk et al., 2004).

Currently, only a handful of studies (e.g., Song and Saraswathy, 2007; Brook and Ben-Dor, 2011; Toda et al., 2012; Lee et al., 2012; Abbas, 2015) have reported the application of vis-NIR spectroscopy in concrete characterization. However, these authors were mostly interested in determining the concentration of chloride in concrete structures. Noticeably, chloride is not the only contaminant in concrete. It is well known that heavy metals (largely from Al₂O₃, SiO₂, Fe₂O₃, MgO, CaO, etc.) are ubiquitous in concrete. Additionally, because of remnants of petroleum-based oil in pre-treated oil-based cuttings (IOGP, 2016), the occurrence hydrocarbon-based contaminants such as TPH concretized (solidified/stabilized) pre-treated oil-based drill cuttings is unavoidable. Recently, Okparanma et al. (2018) suggested the possibility of using vis-NIR diffuse reflectance spectroscopy to determine the contents of heavy metals and TPH in concretized pre-treated oilbased drill cuttings.

This study, therefore, adds to the list of what vis-NIR spectroscopy can (or potentially) do, considering that concrete materials are rarely discussed in NIR circles. The objectives of this study were: (1) to investigate the effects of S/S variables on vis-NIR spectral reflectance of the solidified/stabilized pre-treated oil-based drill cuttings and (2) to adopt the vis-NIR spectroscopic method for the rapid semi-quantitative measurement of TPH, Cu, and Ba contents in the solidified/stabilized matrix to reduce costly delays associated with conventional chemical analytical protocols for enhanced sustainable reuse of the petroleum waste as construction material.

MATERIALS AND METHODS

Study area

This investigation was carried out in Rivers State, Niger Delta

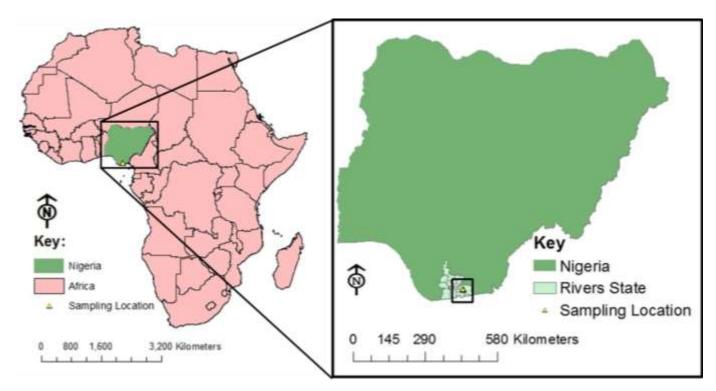


Figure 1. The study area (Datum and Projection: GCS/WGS 1984. Shape file source: ESRI®, CA, USA). (Source: Araka et al., 2019).

region of Nigeria (Figure 1). The Niger Delta region is located in the southern part of the country between latitudes 4° and 6° north of the equator and longitudes 5° and 7° east of Greenwich. The region is mostly a natural wetland covering about 70,000 km² (NDES, 1995) with a predominance of sandy-clay soils (United Nations Environment Programme (UNEP), 2011). The region has a population of over 10 million people and is the hub of Nigeria's crude oil and natural gas activities; the main driver of the country's economy (NDES, 1995). It has been reported that a total of 4,492,057.9 tons of petroleum wastes were generated in only 6 offshore platforms from 2010 to 2013 in Nigeria (Nwokedi et al., 2015). With most of the oil and gas production activities concentrated in the Niger Delta region, it is most likely that a majority of these wastes would come from the region.

Sample collection

Composite samples of the drill cuttings were collected with plastic containers from a drill cutting Treatment Company in the Niger Delta region (Figure 2) and taken to the laboratory for cement-based S/S treatment and subsequent chemical analyses and optical measurement. Sample management followed guidelines in local regulatory framework (DPR, 2002)

Cement-based S/S and reference TPH and heavy metal analyses

The concrete mix design procedure used for the pre-treated oil-based drill cuttings was the Marshal design-procedure for asphalt concrete mixes as described by Igwe et al. (2009). The full account of the cement-based S/S of the pre-treated oil-based drill cuttings has been presented in our previous report (Araka et al., 2019). TPH content of samples was determined according to EPA (1994a)

method 8270 for alkanes and EPA (1994b) method 8270B for PAH by gas chromatography mass spectrometry (GC-MS). TPH extraction from samples, cleanup of the sample extract, GC-MS operating conditions and the type of autosampler installed, TPH quantification method, instrument calibration, and quality control measures adopted are as described by Okparanma et al. (2017). Concentrations of heavy metals in the pre-treated oil-based drill cuttings were analyzed by flame atomic absorption (FLAA) UNICAM-969 Atomic Absorption spectrometry using a Spectrophotometer (AAS) according to APHA (1998) method 3111C. Sample preparation, acid digestion of sample, instrument operating conditions, and instrument calibration are as described in our earlier report (Araka et al., 2019).

Sample optical measurement, development of PLS calibration and validation models

Optical measurement of the samples was carried out with a portable fibre-optic LabSpec2500® vis-NIR spectrophotometer (ASDI, a PANalytical Company, Boulder, CO., USA) of wavelength range 350–2,500 nm in diffuse reflectance mode. Procedures for sample scanning, spectral pre-processing, and establishment of PLS calibration and validation models have been described in full in our previous report (Okparanma et al., 2013a). Pre-processing of sample spectra and PLS regression analysis was carried with the Unscrambler-X® version 10.5 (CAMO Software AS, Oslo, Norway).

Statistical analysis

Statistical indices used to evaluate the quality of the Ba-, Cu-, and TPH-PLS models were the root-mean-square error (RMSE) of cross-validation and prediction and residual prediction deviation (RPD) (Williams and Sobering, 1986; Naes et al., 2002). Coefficient





Figure 2. (a) Heap of pre-treated oil-based drill cuttings at a treatment, storage, and disposal facility in the Niger Delta, Nigeria, and (b) Plastic container used in the collection of pre-treated oil-based drill cuttings. Source: Araka et al. (2019).

of determination (r^2) was used to determine what percentage of the variability in the dataset was explained by the estimated regression line. Model prediction ability was categorized on the basis of the following criteria proposed by Chang et al. (2001): excellent if RPD > 2.0, almost good if $1.4 \le RPD < 2.0$, and unreliable if RPD < 1.4.

RESULTS AND DISCUSSION

Physicochemical characteristics of pre-treated and solidified/stabilized oil-based drill cuttings

This paper will not be discussing the physicochemical characteristics of the pre-treated oil-based drill cuttings as well as the TPH and heavy metal contents of the solidified/stabilized matrix as these have been done in our earlier paper (Araka et al., 2019). As stated, the current paper is dedicated to studying the effects of S/S variables, including drill cuttings content, age of curing, and water-to-cement ratio on vis-NIR diffuse spectral reflectance of solidified/stabilized pre-treated oil-based drill cuttings and development of PLS regression models for predicting TPH, Cu, and Ba in solidified/stabilized pre-treated oil-based drill cuttings.

Vis-NIR diffuse reflectance characteristics of solidified/stabilized pre-treated oil-based drill cuttings

Figure 3 shows the full-wavelength (350-2500 nm) vis-NIR diffuse spectral reflectance curve of solidified/ stabilized pre-treated oil-based drill cuttings. The plot is the average reflectance spectra of the 5% drill cuttings

additions, 6 water-to-cement ratios, and 4 curing ages. In Figure 3, spectral absorption minima of hydrocarbonbased oil can be seen around 1411, 1759, and 2204 nm. Of these, the two absorption minima around 1411 and 2204 nm appear more prominent than the one around 1759 nm. Through spectral matching with existing spectral library in the open literature, the absorption features around 1411 nm in the second overtone region is associated with C-H stretching modes of saturated CH₂ and terminal CH₃ groups, bending modes of R-OH, and vibrational O-H stretching modes of O-H functional group (Figure 3). Whereas saturated CH₂ and terminal CH₃ groups are linked to TPH usually found in hydrocarbonbased oil (Forrester et al., 2013; Webster et al., 2016), R-OH is linked to oxides of metallic ions such as Al, Fe, Mn, or Si (Viscarra Rossel et al., 2006).

As stated, concrete is widely known to contain large amounts of heavy metals predominantly from constituent metal oxides such as Al₂O₃, SiO₂, Fe₂O₃, MgO, CaO, etc. Another equally important contributing source of heavy metals is the mud additives. The O-H functional group is linked to water (Whalley and Stafford, 1992). The absorption around 1759 nm in the first overtone region (Figure 3) is associated with C-H stretching modes of saturated CH2 and terminal CH3 groups and S-H stretching mode of S-H functional group. As stated, saturated CH2 and terminal CH3 groups are linked to TPH. Moreover, the S-H functional group may be linked to sulphate in the calcium aluminate mono-sulphate reported to be formed during the conversion of primary ettringite at the early stages of hydration of the concrete (Portland Cement Association, 2001). The absorption feature around 1950 nm in the first overtone region is

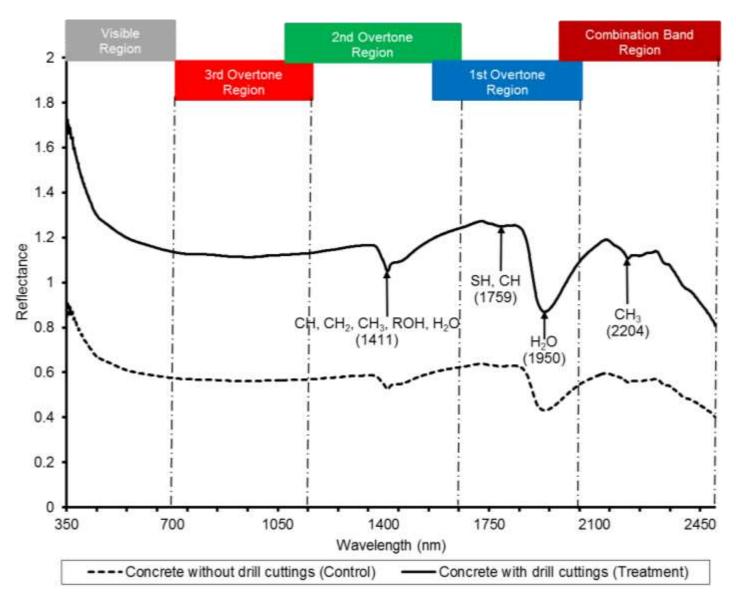


Figure 3. Full-wavelength mean vis-NIR diffuse spectral reflectance curves of the cement-based solidified/stabilized products.

associated with vibrational O-H stretching modes of O-H functional group linked to water while the absorption around 2204 nm in the combination band region (Figure 3) is associated with terminal CH₃ group linked to TPH. These fingerprints suggest that cement-based stabilization/solidification of pre-treated oil-based drill cuttings does not completely eliminate the hydrocarbons and heavy metals in the cuttings.

Effects of drill cuttings content, days of curing, and water-to-cement ratio on diffuse reflectance of solidified/stabilized pre-treated oil-based drill cuttings

Variations of vis-NIR diffuse reflectance of the solidified/

stabilized matrix with drill cuttings content, days of curing (age), and water to cement ratio are shown in Figures 4 to 6, respectively. Reports in the literature suggest that due to age and contamination with other materials such as soil and vegetation, older concretes are darker in colour than newer concretes and absorb more light as well (Fairbarn, 2013). However, concretes produced with pre-treated oil-based cuttings clearly showed the opposite. It is clear that spectral reflectance increased with drill cuttings content (Figure 4), age (Figure 5), and water-to-cement ratio (Figure 6), and vice versa. The increase in reflectance or decrease in absorption with drill cuttings content, age, and water-to-cement ratio might be due to colour-associated changes of the concrete mix material.

Pre-treated oil-based drill cuttings (Figure 2) are light in

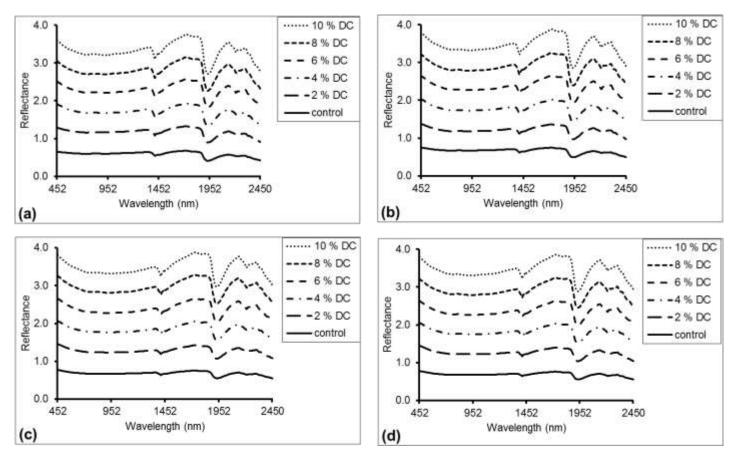


Figure 4. Variation of visible and near-infrared diffuse reflectance with content of pre-treated oi-based drill cuttings added as part replacement for fine aggregate in solidified/stabilized matrix at 0.6:1 water-to-cement ratio after: (a) 7; (b) 14 (c) 21; and (d) 28 days of curing [DC, Drill cuttings].

colour and generally would reflect more light than it would absorb. Even though fine aggregate is lighter in colour than pre-treated oil-based drill cuttings, Figure 4 shows that the concrete produced without pre-treated oil-based drill cuttings had the least reflectance or the most absorption. This suggests that concrete produced without pre-treated oil-based drill cuttings appears darker in colour than concretes produced with pre-treated oil-based drill cuttings when immersed in water in the process of curing. Thus, it is likely that the replacement of fine aggregate with pre-treated oil-based drill cuttings actually lightens the colour of the concrete during curing, thereby increasing the reflectance. This phenomenon may be partly attributed to the effect of water on relics of petroleum-based oil in the pre-treated oil-based drill cuttings. This is because the presence of water has been reported to lighten the colour of relics of petroleum-based oil such as diesel in the soil leading to increased soil reflectance and decreased absorption (Okparanma and Mouazen, 2013b).

Again, the absence of contaminations such as vegetation and dusts from soil in the curing water may be another reason for the observed increase in reflectance

of concrete produced with pre-treated oil-based drill cuttings. More so, soil reflectance is known to decrease as moisture content increases due to natural increase in soil darkness with moisture content (Mouazen et al., 2005).

Quality of PLS regression models for the prediction of TPH and selected heavy metals in cross-validation and validation datasets

In the modelling process, only the 112 crushed stabilized/ solidified samples containing pre-treated oil-based drill cutting were used. The remaining 12 control samples were left out since they did not contain drill cuttings. Table 1 shows sample statistics of PLS regression models for the prediction of TPH, Cu, and Ba in cross-validation and validation data sets. Even though samples of the cross-validation and validation sets were randomly selected, cross-validation dataset means and standard deviations were similar to those of validation datasets. This similarity among cross-validation and validation datasets implied that the prediction models for Cu and Ba

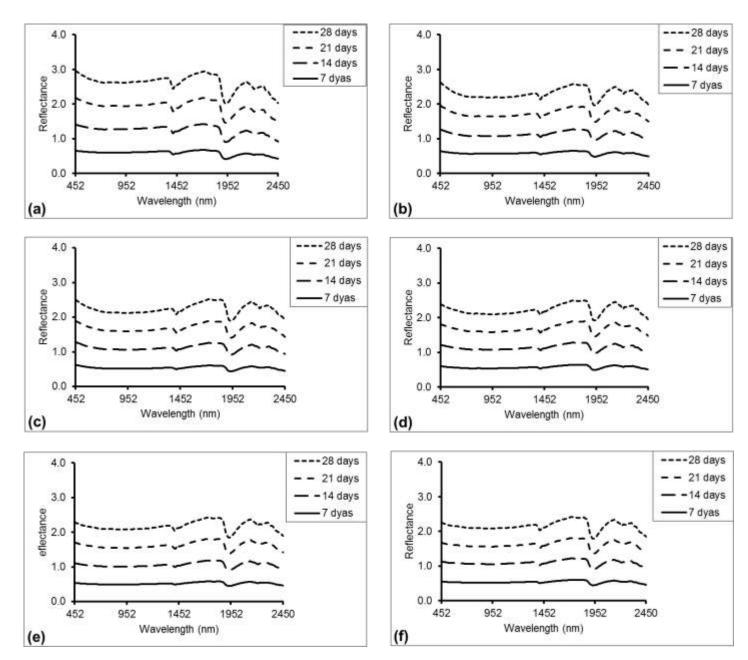


Figure 5. Variation of visible and near-infrared diffuse reflectance with age (in days) of solidified/stabilized matrix produced at 0.6:1 water-to-cement ratio using: (a) 0% (control); (b) 2%; (c) 4%; (d) 6%; (e) 8%; and (f) 10% of pre-treated oil-based drill cuttings as part replacement for fine aggregate.

were not skewed. This situation was a bit different for TPH; the cross-validation dataset mean was similar to the validation dataset mean and the standard deviation of the validation dataset was only one-half that of the cross-validation dataset.

Figure 7 shows the scatter plots of measured versus predicted TPH, Cu, and Ba of the PLS regression models in cross-validation and validation sets for the 112 randomly split treatment samples — 75% for cross-validation and 25% for independent validation. As can be seen in Figure 7, the values of validation ℓ^2 (0.85, 0.89,

and 0.93) and RPD (2.41, 1.97, and 3.45) obtained in this study for TPH, Cu, and Ba predictions respectively indicate the potential of vis-NIR spectroscopy to predict TPH, Cu, and Ba in stabilized/solidified pre-treated oil-based drill cuttings. Similarly, the RMSECV of 7% (TPH) and 6% (Cu) achieved in this study are comparable to the 7% reported by Okparanma (2013b) for PAH prediction in contaminated soils. On the other hand, the RMSECV of 4% for Ba achieved in this study is much better than the 7% reported by Okparanma (2013b) for PAH prediction in contaminated soils. Moreover, on the basis of the RPD

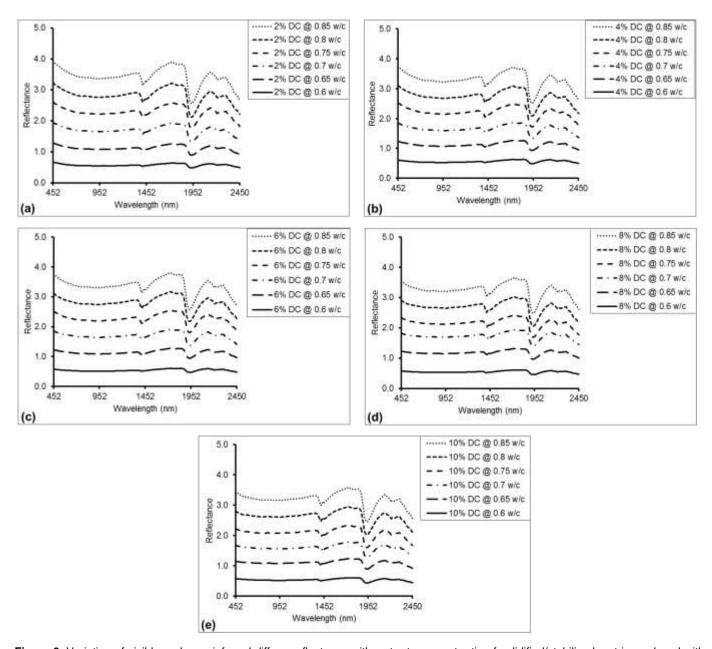


Figure 6. Variation of visible and near-infrared diffuse reflectance with water-to-cement ratio of solidified/stabilized matrix produced with various quantities of pre-treated oil-based drill cuttings as part replacement for fine aggregate after 28 days of curing [DC, Drill cuttings; w/c, Water-to-cement ratio].

Table 1. Sample statistics of partial least squares regression models for the prediction of TPH, Cu, and Ba in cross-validation and validation datasets for 112 crushed solidified/stabilized low-temperature thermally desorbed oil-based drill cuttings using visible and near-infrared spectroscopy.

Number of samples	TPH (mgkg ⁻¹)				Cu (mgkg ⁻¹)				Ba (mgkg ⁻¹)			
	Minimum	Maximum	Mean	SD	Minimum	Maximum	Mean	SD	Minimum	Maximum	Mean	SD
Cross-validation 75% dataset												
84	18.89	167.50	55.88	22.54	3.03	8.23	5.04	0.91	0.08	0.21	0.13	0.02
					Validat	ion 25% datase	et					
28	33.17	89.61	56.49	11.51	2.70	5.59	4.56	0.64	0.08	0.15	0.12	0.02

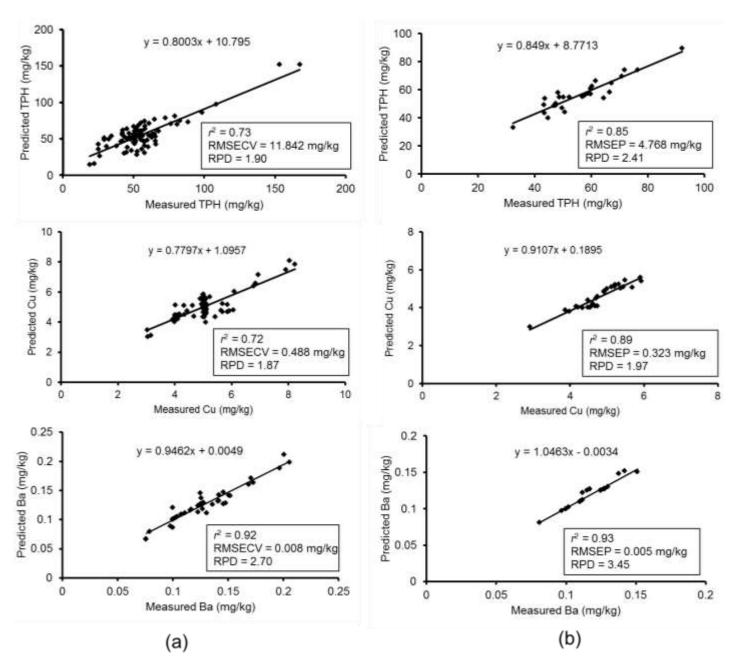


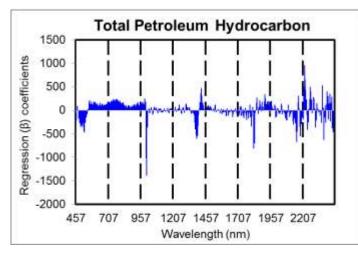
Figure 7. Scatter plots of chemically-measured versus Vis-NIR-predicted values of tph, cu, and ba developed by partial least squares regression analysis with (a) cross-validation 75% dataset and (b) validation 25% dataset. (RMSECV, root-mean-square error of cross-validation; RMSEP, root-mean-square error of prediction; RPD, residual prediction deviation).

values as recommended by Chang et al. (2001), model prediction ability achieved in this study can be categorized as almost good for Cu (validation RPD = 1.97) and excellent for both TPH and Ba (validation RPDs = 2.41 and 3.45, respectively).

Regression coefficients

Figure 8 is the bar plots of regression (β) coefficients

against wavelength from the PLS regression analysis using combined pre-processed vis-NIR spectral data and reference chemical data with 112 calibration samples for TPH and Cu, respectively. Regression coefficients plots are widely used for the identification of important wavelengths for the prediction of pertinent sample properties. The intensity (in absolute terms) of the β coefficients indicates how important the X-variables are in the regression model. Variables with large β coefficient play a key role in the model, with a positive coefficient



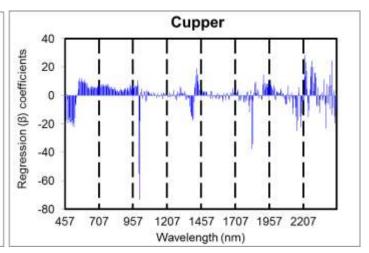


Figure 8. Bar plots of regression (β) coefficients derived from the partial least squares regression analysis using visible and near-infrared diffuse reflectance spectra of stabilized/solidified pre-treated Nigerian oil-based drill cuttings.

showing a positive link to the response and a negative coefficient showing a negative link (CAMO, 2016). As can be seen in Figure 8, both positive and negative β coefficients are present throughout the modelling wavelength range of 457-2450 nm. From Figure 8, it is clear that the intensities of the β coefficients vary considerably in magnitude for TPH and Cu models; which suggests that the β coefficients for the two variables are not homogeneous. However, there are striking similarities in the positions of important wavelengths, which indicate a similarity in the composition of the solidified/stabilized matrix. Larger β coefficients were observed for TPH (957.54-1391.01) compared with Cu (28.35-73.28), which reflect the relative concentrations of the two contaminants in the solidified/stabilized matrix. In the bar plots, positive β coefficients can be seen around 1411 and 1950 nm while negative β coefficients can be seen around 1759 and 2204 nm. The positions of these spectral signatures are exactly the same with those observed earlier in the reflectance curve shown in Figure 3.

Conclusions

applicability of vis-NIR diffuse reflectance spectroscopy in the rapid semi-quantitative measurement of the level of TPH ,Cu, and Ba in cement-based solidified/stabilized pre-treated oil-based drill cuttings was investigated vis-à-vis the effects of S/S variables on spectral reflectance of the solidified/stabilized matrix. Results showed that vis-NIR spectral absorption minima of hydrocarbon-based oil were observed around 1411, 1759, and 2204 nm in the second overtone, first overtone, and combination band regions respectively. Absorption minimum of oxides of metallic ions was observed around 1411 nm in the second overtone region. Absorption minima of water were seen around 1411 and 1950 nm in the second and first overtone regions respectively while absorption minimum of sulphate was observed around 1759 nm in the first overtone region. Spectral reflectance of the stabilized/solidified pre-treated oil-based drill cuttings increased with drill cuttings content, days of curing, and water-to-cement ratio; and vice versa. Model prediction ability was almost good for Cu (validation RPD = 1.97) and excellent for both TPH and Ba (validation RPDs = 2.41 and 3.45, respectively); suggesting the likelihood of using vis-NIR diffuse reflectance spectroscopy as a means for rapid semi-quantitative measurement of TPH, Cu, and Ba contents in cement-based solidified/stabilized pre-treated oil-based drill cuttings.

RECOMMENDATIONS

This study has shown the possibility of using vis-NIR spectroscopy for rapid semi-quantitative assessment of TPH, Cu, and Ba levels in solidified/stabilized pre-treated oil-based drill cuttings using only the design mix ratio of 1:2:4 for a grade-20 concrete. Since all the mix ratios were not considered in the current study, it is possible that not all the sources of variability have been taken into account in the modelling process. Therefore, it is recommended that other mix ratios should be explored.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

ACKNOWLEDGEMENTS

Cranfield University, Bedfordshire, England is highly

appreciated for offering Visiting Student admission to the first author (Student Reference: 254371). Special thanks to Prof. A.M. Mouazen (now in Ghent University, Belgium) for recommending the first author for the admission.

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