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Reviewing the literature on the use of near-infrared analysis to improve the sustainable reuse of pre-treated drill cuttings for building

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There are still some problems with the more appealing sustainable reuse of pre-treated drill cuttings as a partial replacement for fine aggregate in concrete for building applications. This review takes a look at the potential of near-infrared spectroscopy, which has recently been used to non-invasively determine chloride concentration in concrete structures, to quickly determine other important physicochemical properties of concrete made with this hazardous waste. There is a study of drill cuttings' characteristics, origin, content, ecological impacts, and management choices. We also go over some of the lessons learned from using near-infrared diffuse reflectance spectroscopy in real-world soil, petrochemical, environmental, and civil engineering applications while highlighting its basic principles. For improved sustainability as a building material, we also provide a framework for fast near-infrared examination of concrete made with pre-treated drill cuttings.

Key words: Characterization, concrete, drill cuttings, diffuse reflectance spectroscopy.

INTRODUCTION

The dangers posed by the drill cuttings that are an inevitable byproduct of crude oil drilling operations have recently risen to the forefront of environmentalists' minds throughout the world. Drilling for crude oil, whether offshore or on land, used to include dumping untreated drill cuttings into the ocean or onto land, respectively. The devastating effects of land and water disposal of untreated drill cuttings on benthic and edaphic plants and animals have been documented in countless records. There are a number of options for treating drill cuttings at the moment; most of this is mostly motivated by the desire to either salvage the solid parts for potential use in farming (Kogbara et al., 2017) or recycle them for use in civil engineering projects (International Association of Oil and Gas Producers, 2016). Reusing hazardous wastes, such as drill cuttings, in civil engineering projects, such as concrete manufacture, relies on one important principle: making sure the finished product is eco-friendly. The long-term viability of our ecosystem is the goal of these rules. The records that are currently available indicate that contain a high concentration of hydrocarbons and heavy metals (Ayotamuno et al., 2007; Okparanma et al., 2007). Some of these impurities persist even after thermal pretreatment of drill cuttings (IOGP, 2016). Further research has shown that pre-treated drill cuttings have potential as a cement or fine aggregate replacement in concrete (IOGP, 2016). First, the concrete is tested for any remaining impurities in accordance with the principles of sustainable reuse of hazardous materials, such as drill cuttings. Only then is it placed to its intended purpose. Traditional methods of characterizing concrete, such as crushing, acid digestion, solvent extraction, and instrumental analysis, are not without their flaws, however. The use of toxic extraction solvents raises occupational health problems, which in turn causes expensive delays, and high analytical expenses are another issue. Perhaps these mysteries are what set off the search for new ways to analyze concrete. There are indications to suggest that near-infrared (NIR) spectroscopy has the potential to be used for the analysis of certain chemical

properties of concrete such as chloride concentration (Abbas, 2015). NIR spectroscopy is well known for its short analytical time, cost-effectiveness, non-invasiveness, portability, and ecological friendliness (Okparanma and Mouazen, 2013a). Besides, NIR spectroscopy has been extensively used in proximal soil sensing (Mouazen et al., 2010) and, on occasion, for the determination of chloride concentration in concrete (Abbas, 2015). However, NIR spectroscopy has never been used to determine the fundamental physical properties and other mechanical properties of concrete (such as compressive strength, permeability, bulk density, etc.). Similarly, NIR spectroscopy has not been used to assess the physicochemical characteristics of pre-treated drill cuttings such as bulk density, moisture content, as well as hydrocarbon and heavy metal contents etc.

The goals of this report were as follows: (1) to examine the source, composition, ecological effects of, and management options for drill cuttings, as well as the practical applications of NIR spectroscopy in soil science and petrochemical, environmental, and civil engineering; and (2) To highlight the possibility of using NIR spectroscopy for rapid characterization of pre-treated drill cuttings and concrete produced with pre-treated drill cuttings as part substitute for fine aggregate to enhance the sustainable reuse of pre-treated drill cuttings as a construction material.

SOURCE AND COMPOSITION OF OILFIELD DRILL CUTTINGS

There will always be dangerous byproducts from drilling for crude oil and gas in the course of oil and gas exploration and production. Nowadays, dealing with these types of trash is among the serious problems with the environment that the petroleum industry faces. The resultant waste stream's volume and chemical are to blame for this. The solid part of the waste stream is made up of drill cuttings, which are ground rocks, sands, and clays that are released from the well as it drills through geological formations. The liquid part is made up of produced water, crude oil, spent drilling muds, and additives. (Lyons, 1996; Department of Petroleum Resources (DPR), 2002; Norwegian Oil Industry Association (OLF), 2003; Alberta Environment, 2005). There isn't enough room in this article to go into detail into the liquid phase, but readers who are interested may find more in works like Lyon (1996). According to Okparanma and Ayotamuno (2008) and Okparanma et al. (2010), drill cuttings are known to include heavy metals and petroleum hydrocarbons, two types of inorganic and organic contaminants, respectively. The reason for this is because the chemistry of the drill cuttings is affected by the insitu chemical composition of the patented drilling mud and mud additives (Wills, 2000). Common drilling muds include oil-based mud (OBM), water-based mud (WBM), and synthetic-based mud (SBM). Figure 1 displays the approximate weight percent compositions of the most prevalent drilling muds (IOGP, 2016). The composition of WBMs is often brine/water combined with bentonite (clay)/polymer and barite, as seen in Figure 1a. These mud systems are believed to be less hazardous to the environment than others. Non-aqueous drilling fluids (NADFs) are a common term for both oil-based and synthetic base fluids, which are often combined with brine and barite (Figure 1b). The basic fluids used in SBMs are artificially made to act similarly to drilling fluids obtained from petroleum. They are less toxic to the environment and supposedly biodegrade quickly compared to OBMs' non-aqueous base fluids (Lima, 1996). Crude oil, diesel fuel, common mineral oils, and other petroleum fractions containing polycyclic aromatic hydrocarbons (PAHs) make up the non-aqueous basic fluids (Lyons, 1996). Of all mud systems, OBMs are the most environmentally damaging because of these, which are responsible for their toxicity (Lima, 1996). Nigeria (DPR, 2002), Norway (OLF, 2003), Indonesia (Permata and McBride, 2010), and a few other nations have therefore banned OBMs. Nevertheless, OBMs are often chosen over other mud systems, particularly for deep drilling, because to their unique drilling characteristics, such as greater temperature stability, lubricity, and hole stabilizing abilities (Caenn and Chillingar, 1996). It turns out that several oil-producing nations are still using OBMs, albeit in less quantities, which is harmful to both humans and the environment (Okparanma et al., 2010). In addition to non-aqueous base fluids, NADFs include additives like barite and other weighing agents, as well as thinners and emulsifiers (Figure 1b).

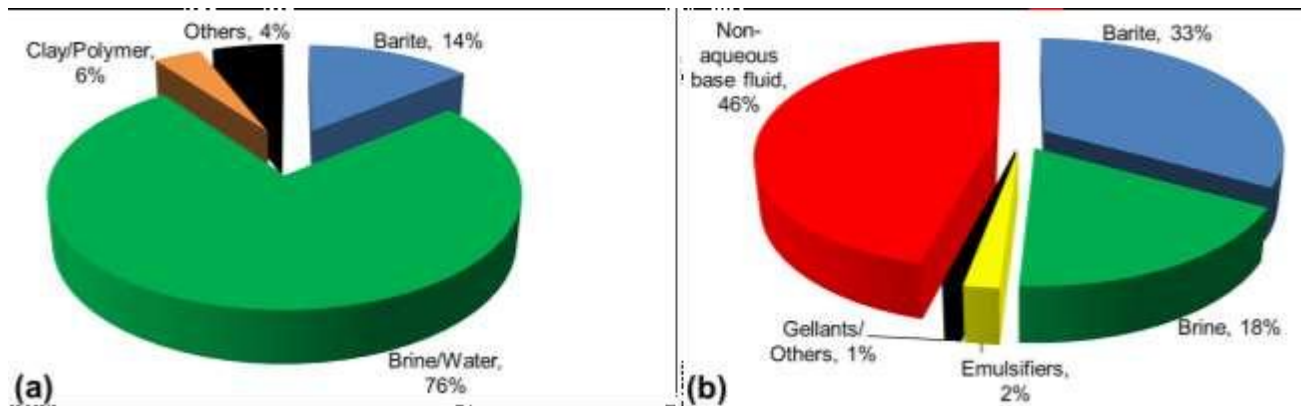


Figure 1. Approximate percentage weight composition of typical: (a) Water-based mud; and (b) Non-aqueous drilling fluids. Source: IOGP (2016).

for the heavy metal content of the drilling muds. Table 1 shows the reported physicochemical characteristics of untreated and thermally treated oil-based drill cuttings and the common mud systems. As can be seen in Table 1, the drill cuttings still contain traces of heavy metals and hydrocarbons even after thermal treatment. This underpins the need for proper physicochemical characterization of concretes produced with thermally treated (also known as pre-treated) drill cuttings before putting them to the intended use.

ECOLOGICAL EFFECTS OF DRILL CUTTINGS

The ecological effects of drill cuttings when disposed of on land or sea without prior treatment have been widely documented in the open access literature (Ellis et al., 2012; Zamora-Ledezma and García, 2013; Larsson et al., 2013; Durgut et al., 2015; Nilssen et al., 2015). In a recent review, Ellis et al. (2012) revealed a loss of benthic biodiversity and suspension-feeding communities due to discharge of drilling waste from oil and gas platforms. Zamora-Ledezma and García (2013) reported that drill cuttings are extremely toxic for some species of tropical grasses including *Urochloa decumbens* and *Urochloa maxima*. There have also been reported lower growth rates of cold-water coral *Lophelia pertusa* when exposed to water-based drill cuttings for 12 weeks (Larsson et al., 2013). Recently, while assessing the potential impact of water-based drill cuttings on two deep-water calcareous red algae, namely; *Mesophyllum engelhartii* and *Lithothamnion* sp., Nilssen et al. (2015) observed an occasional risk for severe impact on the calcareous algae. Therefore, effective management of drill cuttings becomes a consequent necessity, not least because some drill-cuttings contaminants such as PAHs are non-threshold carcinogens known to possess some genotoxic potential (Contaminated Land: Applications in Real Environments (CL:AIRE), 2010).

DRILL-CUTTINGS MANAGEMENT

Figure 2 shows a schematic of possible management options for drill cuttings, modified after International Association of Oil and Gas Producers (IOGP). As pointed out, one of the two key objectives that need to be addressed in the management of drilling waste is to comply with environmental regulations (IOGP, 2016), which are meant to protect ecological health.

Solids control, cuttings re-injection, and sea and land disposal

As can be seen in Figure 2, management of the waste stream from drilling operation starts from the shale shaker, which separates the solid (drill cuttings) from the liquid phase. The liquid part is recycled after pretreatment by desanding, desilting, or centrifuging. The drill cuttings may be re-injected into a re-injection well in suitable formations after slurrification, disposed of on land or sea. As stated, cuttings re-injection, sea and land disposal of

drill cuttings without prior treatment have negative ecological impact associated with them. This explains why it is often recommended that drill cuttings are subjected to further secondary treatment before final fate. The current review focused on the reuse of pre-treated drill cuttings as construction material, particularly concrete. However, this could not be discussed in isolation of other treatment options for drill cuttings, especially secondary treatment technologies.

Secondary treatment technologies for drill cuttings

Secondary treatment technologies for drill cuttings may be broadly categorized into two, namely; the non-biological and biological treatment technologies (Figure 2).

Table 1. Physicochemical properties of untreated and treated oil-based drill cuttings and the two common mud systems.

Property	Oil-	ill cuttings	NADF ^{d, e}	WBM ^{d, e, f}	
	based dr	Treated ^b			
	Untreated ^{a, b, c}				
pH	7.1	NR	NR	11.7	
Sodium adsorption ratio (meq 100 g ⁻¹)	5.3	NR	NR	NR	
Exchangeable bases (meq 100 g ⁻¹)	6.2	NR	NR	NR	
Cation exchange capacity (meq 100 g ⁻¹)	6.8	NR	NR	NR	
Base saturation (%)	99	NR	NR	NR	
Exchangeable aluminium (%)	1.2	NR	NR	NR	
Electrical conductivity (mS cm ⁻¹)	0.8	NR	NR	NR	
Salinity (g l ⁻¹)	0.9	NR	22.5 (%)	NR	
Oil and grease (% weight)	13.7	NR	NR	NR	
Moisture content (w/w %)	20.5	0.3	NR	NR	
Dry matter (g/kg)	561 - 798	990 - 997	NR	NR	
Volatile dry matter (g/kg)	51.8 - 90.2	40.5 - 91.9	NR	NR	
Sand (%)	NR	5.4 - 29.1	NA	NA	
Silt (%)	NR	19.4 - 75.4	NA	NA	
Clay (%)	NR	19.2 - 64.7	NA	NA	
HM (mg/kg):	Aluminium (Al)	40,400	NR	52,000	10,800
	Arsenic (As)	2.9 - 8.5	4.0 - 8.4	NR	NR
	Barium (Ba)	270 - 8100	160 - 6900	487,000	720 - 449,000
	Iron (Fe)	21,900	NR	76,300	0.002 - 27,000
	Lead (Pb)	12 - 27	21 - 30	NR	NR
	Cadmium (Cd)	0.06 - 0.36	0.08 - 0.22	0.39 - 12	0.16 - 54.4
	Copper (Cu)	15 - 52	25 - 180	NR	NR
	Chromium (Cr)	19 - 26	22 - 28	190 - 1,350	0.1 - 5,960
	Mercury (Hg)	0.017 - 0.073	0.025 - 0.087	NR	0.017 - 10.4
	Nickel (Ni)	16 - 25	17 - 31	NR	NR
	Zinc (Zn)	44 - 160	55 - 200	160 - 2,100	0.06 - 12,270
	Naphthalene	<0.0005 - 0.6	<0.0005 - 0.25	NR	NR
	Acenaphthylene	<0.0005 - 0.76	<0.0005 - 0.011	NR	NR
	Acenaphthene	<0.0005 - 3.8	<0.0005 - 0.18	NR	NR
	Fluorene	<0.0005 - 0.17	<0.0005 - 0.024	NR	NR
Phenanthrene	<0.0005 - 0.26	<0.0005 - 0.17	NR	NR	

PAH (mg/kg):	Anthracene	<0.0005 - 0.023	<0.0005 - 0.023	NR	NR
	Fluoranthene	<0.0005 - 0.056	<0.0005 - 0.045	NR	NR
	Pyrene	<0.0005 - 0.093	<0.0005 - 0.091	NR	NR
	Benzo[a]anthracene	0.003 - 0.042	0.006 - 0.035	NR	NR
	Chrysene	<0.0005 - 0.041	<0.0005 - 0.057	NR	NR
	Benzo[k]fluoranthene	<0.005 - 0.025	0.004 - 0.045	NR	NR
	Benzo[a]pyrene	<0.005 - 0.013	0.002 - 0.029	NR	NR
	Indeno[1,2,3-cd]pyrene	0.003 - 0.07	0.004 - 0.07	NR	NR
	Dibenzo[a,h]anthracene	<0.005 - 0.01	0.003 - 0.02	NR	NR
	Benzo[ghi]perylene	<0.0005 - 0.01	0.004 - 0.1	NR	NR

HM, Heavy metals; NADF, Non-aqueous drilling fluid; PAH, Polycyclic aromatic hydrocarbons; NA, Not applicable; NR, Not reported; WBM, Water-based mud. ^a Data from Zamora-Ledezma and Garcia (2013); ^b Data from Vik et al. (2014); ^c Data from Deeley (1990); ^d Data from Health and Safety Executive (2000); ^e Data from Neff et al. (2000); ^f Data from Safi et al. (2016).

Non-biological treatment technologies

Drill cuttings are treated by non-biological methods such as drying (Schlumberger, 2016), solidification and stabilization (Akinlade et al., 1996; Shaffer et al., 1998; Fleming, 2000; Leonard and Stegemann, 2010; Opete et al.,

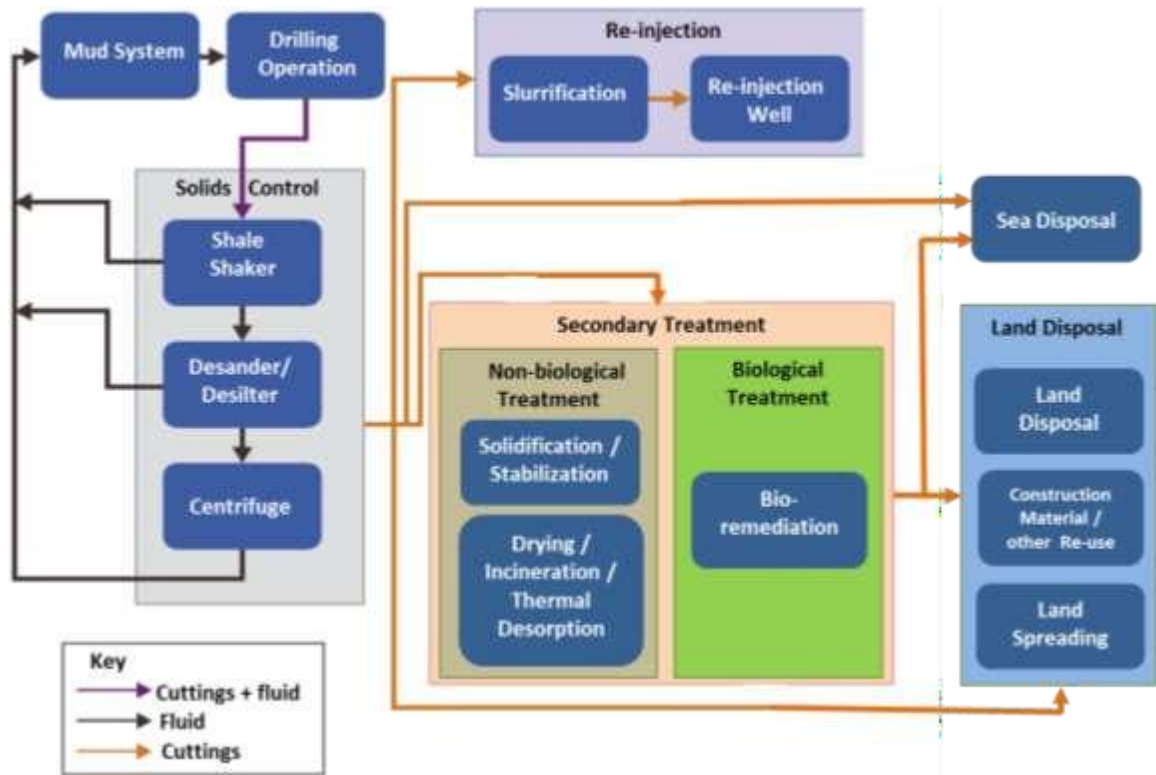


Figure 2. Schematic of possible management options for drill cuttings.
 Source: IOGP (2016): reproduced after the permission of IOGP but with slight modification.

a compressive strength of 1,377.14 kN/m² was reported. However, in a similar exercise conducted by ChevronTexaco, Fleming (2000) reported that eight different commercial additives tested failed leachability tests due to sudden breakdown of the matrix or failure of the fixing agents to keep the drill-cuttings constituents from leaching to the environment over a long period of time. Studies carried out in Southern Louisiana University indicated that drill cuttings stabilized in a silica matrix had a pH greater than 11 and did not support plant growth when land applied (Shaffer et al., 1998). Again, Leonard and Stegemann (2010) used Portland cement mixed with high carbon-power plant fly ash (as a sorbent for organic contaminants) to stabilize drill cuttings. They reported that the drill cuttings and high carbon-power plant fly ash addition both reduced the unconfined compressive strength of the S&S products. Moreover, in the S&S treatment of synthetic drill cuttings with ordinary Portland cement (OPC), Opete et al. (2010) reported that concentrations of arsenic, mercury, and silver in the resulting concrete were reduced below local regulatory values. However, they observed that cadmium, chromium, and zinc contents were higher than the local permissible limits. Similarly, Ghasemi et al. (2017) used OPC (Type I 42, 5) and modified clay (Claytone[®] 40) mixes for the S&S treatment of oil-based drill cuttings. They observed that at a given clay content, an increase in cement content lowers the leaching rate; but, at a given amount of cement, increasing the clay content does not necessarily reduce the rate of leaching.

Thermal treatment: Thermal treatment involves high temperature heating of drill cuttings in order to reclaim or completely destroy them, which results in immobilization of inorganic metals and salts and overall reduction in drill cuttings volume (Bansal and Sugiarto, 1999). There are two main categories of thermal treatment, namely; incineration and thermal desorption.

Incineration: Incineration is the combustion of wastes at high temperatures (1,200 to 1,500°C), which is aimed at converting them into ash that is less bulky, non-hazardous, or less hazardous than they were before incineration (Morillon et al., 2002). An incineration method and apparatus for treatment of oilfield wastes was patented by Dugat (1988). The pre-treated wastes are dried and incinerated to a char solids condition. The char solids and the heating gases are cooled by heat exchange to provide hot air for combustion in the dryer-incinerator, thereby saving heating fuel cost. The gases are then scrubbed to remove contaminants and the condensed water-hydrocarbon phases are separated for reuse or for further treatment for recovery (Dugat, 1988). Since the main objective of thermal treatment of drill cuttings is not to char them but to reclaim the drill cuttings for possible land application or other forms of reuse, incineration method is less frequently used in the treatment of drill cuttings. Nonetheless, incineration has greater application in the treatment of medical wastes (Adeniran, 2003), which is outside the scope of the current review.

Thermal desorption: Thermal desorption refers to the indirect heating of drill cuttings to vaporize volatile and semi-volatile compounds without incinerating the drill cuttings (Figure 3). It is important to state that unlike incineration, thermal desorption does not produce ash as its end product since the aim is not to completely destroy



Figure 4. Heap of pre-treated drill cuttings from the anaerobic thermal desorption unit (ATDU) of a commercial treatment facility in the Niger Delta, Nigeria.

the cuttings by converting them to ash but to recover the oil-free cuttings. This distinction is important because thermally desorbed drill cuttings apparently have often been referred to as drill-cuttings ash in some literatures.

Low-temperature thermal desorption units (250 to 350°C) are used to remove light aliphatics and aromatics (e.g., benzene, toluene, ethylbenzene, and xylenes); while high-temperature thermal desorption units (up to 520°C) are used to remove heavier aromatics such as PAHs (Owens et al., 1993). Thermal desorption includes (among others) thermal phase separation (DWMIS, 2000), which consists basically of five subsystems, namely; the pretreatment unit, the anaerobic thermal desorption unit (ATDU), the solids handling unit, the vapour recovery unit (VRU), and the water treatment unit (WTU) (Zupan and Kapila, 2000). At the pretreatment unit, the drill cuttings are screened to remove foreign and oversized materials (>50 mm in diameter) before delivery to the ATDU. The drill cuttings are heated indirectly in the ATDU under an oxygen deficient condition to a temperature equal to the boiling point of the hydrocarbons, usually about 220°C but sometimes up to 520°C. At this temperature, the hydrocarbons are volatilized and separated from the drill cuttings. The processed drill cuttings exiting the ATDU are received in the solids handling unit where they are cooled and kept in a storage area (Figure 4).

The gases (mixture of water vapour and gaseous hydrocarbons) exiting the ATDU enter the VRU housing the scrubbers. The condensed liquids and recirculated quenched water are then sent to the WTU where oil is separated from water. However, because of the indirect heating system of the ATDU, large heating surfaces that consume large amounts of energy and space are needed (Zupan and Kapila, 2000). Moreover, the operating temperature of 520°C for high temperature TDUs could lead to thermal degradation and decomposition of the residual drill cuttings

(DWMIS, 2000). It has also been reported that thermal desorption is associated with high cost implications (Bansal and Sugiarto, 1999), environmentally threatening consequences such as high-level occupational hazard resulting from heat and fugitive dust emissions from the ATDU (DWMIS, 2000), and costly delays due to the length of time spent in the separating units (Ball et al., 2012). Furthermore, using low temperature thermal desorption to treat oil-based drill cuttings containing up to 21.8% of oil; Onwukwe (2014) reported that only 0.33% of the oil were left in the drillcuttings after 45 min of treatment. They suggested that thermal desorption can remove a reasonable amount of oil from oil-based drill cuttings.

Microwave-assisted heating: In microwave-assisted heating of materials, energy is transferred directly to substances. This causes every single element in the material to be heated instantly through molecular or ionic vibrations under the effect of an oscillating magnetic or electric field (Mutyalu et al., 2010; Robinson et al., 2009). Although, microwave-assisted heating technology is not applicable to every material, it is becoming increasingly attractive in the treatment of oilfield drill cuttings because of the presence of microwave-absorbing materials like carbon and water in drill cuttings (Robinson et al., 2008). In one study, Robinson et al. (2009) used a pilot-scale microwave heating system to remediate oil-based drill cuttings, reducing the residual oil contents to less than 1% of its initial amount. However, during the microwave remediation of drill cuttings, Petri et al. (2015) reported that a high initial drilling fluid content in drill cuttings reduced the oil removal efficiency of the microwave heating method. Generally, microwave heating method has not been commercialized on a large scale as yet due to the huge financial implications for initial setup; as only a few small-scale commercial applications of the technology have been reported in the literature (Mutyalu et al., 2010).

The enumerated challenges associated with the non-biological methods prompted the shift of emphasis to biological treatment technologies like bioremediation, which according to several literatures has high potential for restoring polluted media with least negative impact on the environment at relatively low cost.

Biological method

Bioremediation refers to the process of using microorganisms like fungus and bacteria to facilitate the natural breakdown of waste materials polluted with hydrocarbons into harmless byproducts (Atlas and Bartha, 1972). Complete decontamination of hydrocarbon-contaminated matrixes may take up to two years when performed alone during bioremediation (KMC Oiltools, 2005), prompting the development of new approaches to speed up the process. These strategies are broadly categorized into ex-situ and in-situ techniques including (but not limited to) composting with decomposing organic materials in biopiles and/or windrows (Coulon et al., 2010), biostimulation using organic or inorganic fertilizers (Ayotamuno et al., 2009), bioaugmentation using bacteria isolates (Okparanma et al., 2009), mycoremediation using fungi species (Okparanma et al., 2011), remediation by enhanced natural attenuation (RENA) (Ebuehi et al., 2005), and onsite exsitu RENA (Okparanma et al., 2017). In a controlled environment, For the purpose of bioremediation, drill cuttings have been composted using farmyard manure and poultry droppings, bioaugmented with two bacterial strains (*Pseudomonas aeruginosa* and *Bacillus subtilis*), and mycoremediated with white-rot fungus (*Pleurotus ostreatus*) (Okparanma et al., 2009, 2011; Ayotamuno et al., 2009, 2010). Some have raised concerns about the possibility of groundwater contamination and the limited availability of land due to the large-space requirements associated with field-scale RENA treatment of drill cuttings in biopiles or windrows (Ayotamuno et al., 2010; Maduekwe et al., 2016). The ability of laboratory-prepared microbes to adapt to field conditions is an overarching issue, and there are three associated huge financial burdens with bioaugmentation that have been reported to be significant (KMC Oiltools, 2005). Another issue is the large volume of bio-preparation of microbial consortiums needed for field use. To our knowledge, no published research on the bioremediation of drill cuttings using mycoremediation has been conducted on a large scale. In the meantime, studies are looking at ways to reuse pre-treated drill cuttings in a sustainable and useful way.

Putting Pre-Treated Drill Cuttings to Use in the Building Process The world over, the costs of cement as well as fine and coarse aggregates is quite high due to rising inflation. It is now necessary to investigate other, less expensive building materials that might serve the same purpose. According to Chen (2007), Allen et al. (2007), IOGP (2016), and others, pre-treated drill cuttings are starting to gain recognition as a cement substitute in concrete, aggregate in cold-mix asphalt for road pavement construction, and fill material for road construction. In their 2007 study, Chen et al. used drill cuttings to make

bricks and concrete, partially replacing cement in the process. According to their findings, the bricks' compressive strength and permeability were sufficient for construction. However, the resulting concrete showed variable compressive strength and permeability and was sensitive to the mix design (Chen et al., 2007). The use of drill cuttings as aggregate in cold-mix asphalt for the building of road pavements was documented by Allen et al. (2007). Table 1 shows that the high hydrocarbon and moisture levels of the drill cuttings were initially restrictions that were eventually overcome (Allen et al., 2007). It is worth noting that there is ample documentation in the literatures comparing various drill cuttings management solutions in terms of mobility, relative cost, commercialization, environmental consequences, benefits, and limits.

For further details, interested readers might refer to IOGP (2016).

Concrete: Admixture proportions and varieties of concrete

The majority of building and heavily travelled pavement structural components are made of concrete (Igwe et al., 2009). A typical recipe for concrete calls for water, fine and coarse aggregates, and Portland cement; the mixture is then left to cure for at least 28 days (Adewole et al., 2015). It is important to state that the curing period of 28 days is only limited to some laboratory-scale studies (Anonymous Reviewer). The type of loading to which the concrete is subjected when in normal use is determined by its grade/strength class, which is dependent on the mix ratio. While the most common mix ratio is the 1:2:4 (cement–fine aggregate–large aggregate); other mix ratios such as the 1:1.5:3 and 1:1:2 have also been tested (Adewole et al., 2015). Concrete grade/strength class indicates the minimum compressive strength of concrete, which is taken as the 28-days crushing strength of concrete cubes ($150 \times 150 \times 150$ mm) or cylinders (150 dia. \times 300 mm) (BSI, 2004). According to BSI (2004), the concrete grade/strength class of C20/25, for example, should have at least a cylinder and cube crushing strengths of 20 and 25 MPa, respectively. This concrete grade/strength class is largely recommended in the design of reinforced concrete columns, beams, and slabs for use under mild exposure condition (Mosley et al., 2007). To achieve the minimum compressive strength of concrete recommended for use under a given loading condition, it is also important that the appropriate grade of cement is used in the mix design to avoid structural failure (Adewole et al., 2015). But, prior to using concrete made with pre-treated drill cuttings as partial substitute either for cement or fine aggregate to the intended use, it is first analyzed to check if it is fit for purpose. This is in relation to compliance with environmental guidelines and recommended minimum compressive strength of concrete. More often than not, conventional methods for the determination of the physicochemical properties of the resulting concrete have a variety of challenges, which call for alternative approaches.

Conventional methods of concrete characterization and their challenges

Determination of the organic and inorganic contents of concrete such as hydrocarbons, chloride, and heavy metals are based on laboratory wet-chemistry analyses. The protocols generally adopted include slicing and crushing of the concrete, acid digestion of the crushed concrete, solvent extraction and clean-up of the extract, and analysis of the extract either by gas chromatography or chemical titration. For hydrocarbon analysis, a gas chromatograph coupled to either a flame ionization detector or mass selective detector is used according to USEPA method 8270 (USEPA, 1994). For heavy metal analysis, an atomic absorption spectrophotometer is used according to APHA method 3111C (APHA, 1998). Both the water extractable and the acid soluble chlorides are analyzed using potentiometric titration with silver nitrate according to AASHTO method T26097 (AASHTO, 2005). According to a recent review (Okparanma and Mouazen, 2013a), solvent extraction is both time-consuming and associated with ecological health risks because extraction solvents and stock solutions often used in the extraction process and a priori instrument calibration respectively are carcinogenic. In the same vein, analytical cost per sample for hydrocarbons using gas chromatography-mass spectrometry (GC-MS) is very high and the process is slow in addition to the need for an experienced operator to run the analysis and later process the data. This implies that the GC-MS method is a major economic burden to researchers when dealing with a large sample size. Moreover, the total dependence of the Cube Crushing Machine on electricity during the determination of compressive strength of concrete cubes in the laboratory is a huge limitation especially in areas where regular power supply is unreliable. Again, since the Cube Crushing Machine is not portable, it will require lots of labour and man-hour to determine the compressive strength of a large number of concrete cubes. Therefore, there is a need to evolve a rapid, cheap, portable, and ecologically sound approach for the determination of the physicochemical characteristics of concrete produced with pre-treated drill cuttings as fine aggregate.

NEAR-INFRARED (NIR) SPECTROSCOPY

As stated, because of the challenges associated with wet chemistry methods of concrete analysis, alternative methods are now emerging. One of these alternatives is the optical method based on visible (vis), NIR, and mid- infrared (MIR) spectroscopy. Compared to MIR, NIR is more robust for in-situ measurement and less sensitive to moisture. However, as subsequently discussed, the current application of vis-NIR spectroscopy in concrete analysis has been largely focused on the determination of chloride concentrations in concrete structures. This is despite the fact that it is well known that chloride is not the only contaminant usually found in concrete (IOGP, 2016). Meanwhile, to drive home the aim of this review, it would be worthwhile to briefly describe the fundamental principles of NIR spectroscopy. Principles of NIR spectroscopy

Figure 5 shows the different regions in the electromagnetic

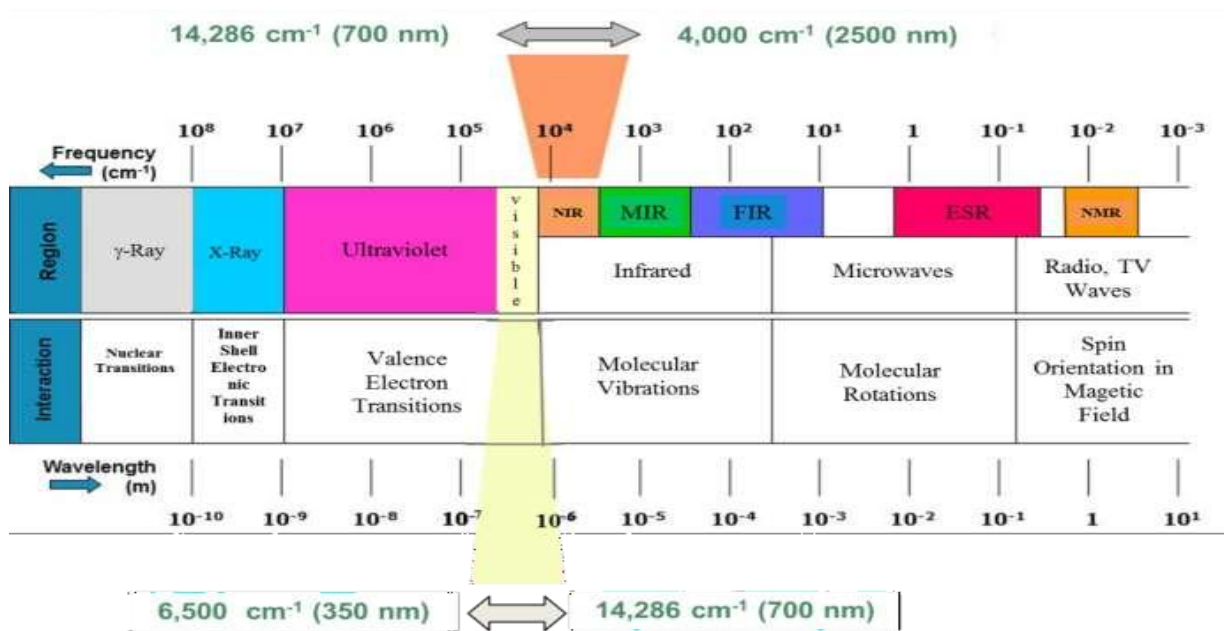


Figure 5. The electromagnetic spectrum. (Source: Bruker Optik GMBH <https://www.bruker.com/products/infrared-near-infrared-and-raman-spectroscopy.html>; reproduced with permission of Bruker Optik GMBH but with slight modification) NIR = Near-Infrared, MIR = Mid-Infrared, FIR = Far-Infrared, ESR = Electron Spin Resonance, NMR = Nuclear Magnetic Resonance.

spectrum. In the current review, only the vis and NIR regions are of interest because of their versatility in the analysis of a wide range of samples including both solids and liquids such as cereals, soils, powders, oils, etc.; all in one single stand-alone device. In the electromagnetic spectrum, the vis region is around 350–700 nm while the NIR region is around 700–2500 nm (Figure 5). Thus, vis- NIR spectroscopy covers the region around 350–2500 nm. The use of vis-NIR spectroscopy in soil physicochemical analysis both spatially and temporally is now an all too common practice. Vis-NIR spectroscopy is based on the interaction of electromagnetic waves (radiation) with sample

constituents (matter). The principles of vis-NIR diffuse reflectance spectroscopy involving the interaction of electromagnetic waves with sample constituents is shown in Figure 6. Unlike specular reflected light (related to mirror-like surfaces), diffuse reflected light (related to powder-like material such as soils) interacts with the constituents of the samples; and its spectrum on a detector provides qualitative and/or quantitative information about the sample. Therefore, the spectrum of a sample is unique — like a fingerprint; and is used to identify the sample. The detector is equipped with an array of Michelson interferometers and filters. There are different types of detectors, including diode arrays, monochromators, etc.

Origin of NIR spectroscopy

According to Davies (2005), NIR was discovered by William Herschel in 1800. With inputs from Ampere in 1835 and James Maxwell in 1864, the first NIR spectra were measured in 1881 by Abney and Festing. Following the successes recorded by Abney and Festing by accurately predicting that spectral absorptions were related to the chemical composition of liquids, Coblenz in 1905 published the spectra and corresponding wavelengths (1000–10,000 nm) of a large list of compounds. This paved the way for vibrational spectroscopy of molecules in the MIR (2500–50,000 nm) region. Then in 1960 Karl Norris of the United States Department of Agriculture showed the effectiveness of the NIR region in quantitative analysis of agricultural samples.

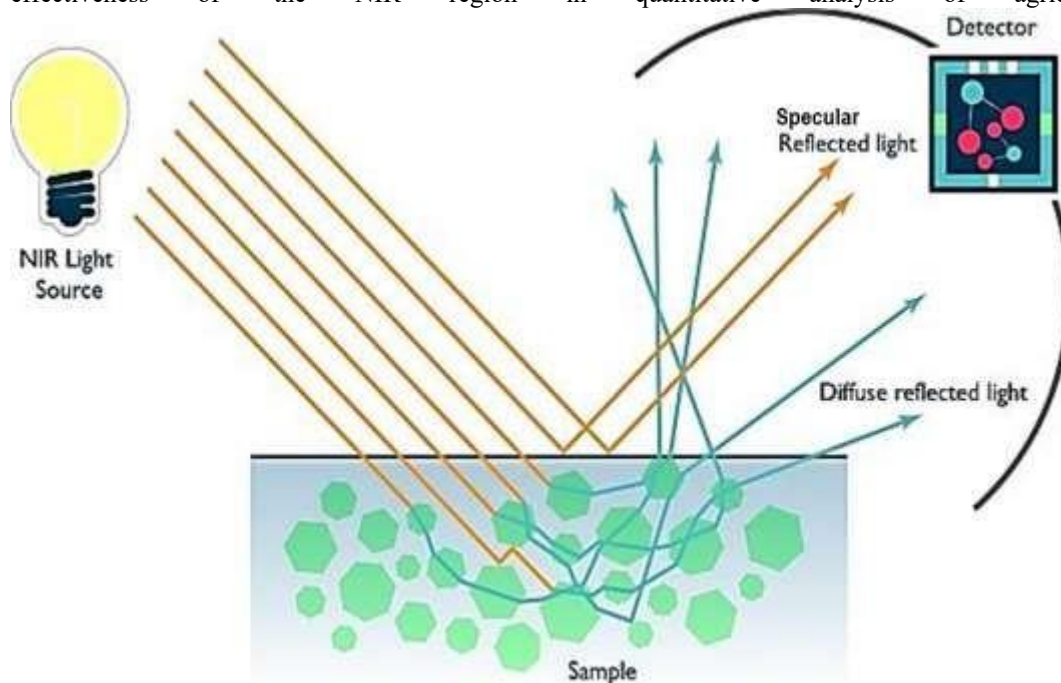


Figure 6. Principle of near-infrared diffuse reflectance spectroscopy (Source: Pulp & Paper, Canada <http://www.pulpandpapercanada.com>; reproduced with permission of Pulp & Paper, Canada).

Scattering and absorption of energy (light) by sample in the vis-NIR region

Scattering and absorption of light by solid samples in the vis-NIR region are two important phenomena that influence the measurement (in diffuse reflectance mode) of both physical and chemical properties of solid samples.

(a) Scattering of light by sample: Light scattering by solid samples (Figure 6) is associated with the physical

characteristics of the sample (e.g., particle size), as well as the chemical characteristics such as hydrocarbon content (among others). When light is incident on a solid sample, the light is scattered in different directions depending on the geometry and size of the solid granule, as well as the wavelength and frequency of the incident light. If the light scattering is elastic, it is referred to as Rayleigh scattering (Kaminow and Li, 2002). Rayleigh scattering intensity is largely dependent on the particle size of the sample; it is proportional to the sixth power of the particle diameter and inversely proportional to the fourth power of the wavelength of light (Kaminow and Li, 2002). Therefore, Rayleigh scattering intensity is stronger around 450 nm (blue colour) than 680 nm (red colour) in the vis region; suggesting that the larger the particle size of the sample is, the stronger is the Rayleigh scattering intensity and vice versa. On the other hand, if the light scattering is inelastic, it is referred to as Brillouin or Raman scattering. Although central to Brillouin and Raman scattering is the interaction of light with the constituents of the solid sample, both types of light scattering are different in the way the light interacts with the solid sample (Kaminow and Li, 2002). Raman scattering may be used to assess the vibration of Raman-active molecules such as the PAHs, as well as for identifying species. According to a recent review (Okparanma and Mouazen, 2013a), qualitative and quantitative applications of Raman spectroscopy is widespread.

(b) Absorption of light by sample: Absorption of light by solid samples is linked to both the physical characteristics (e.g., colour) and chemical characteristics (e.g., water, hydrocarbons, amines, clay minerals) of the sample. In the vis region of the electromagnetic spectrum (Figure 5), absorption of light by solid samples is linked to colour and electron excitation. Colour associated changes due to colour absorption bands around 450 nm (blue colour) and 680 nm (red colour) have been linked to changes in spectral geometry of solid samples (Mouazen et al., 2007; Okparanma and Mouazen, 2013b). For instance, soil diffuse reflectance is known to decrease as the soil gets darker in colour due to increased light absorption, and vice versa (Mouazen et al., 2007). Similarly, oil contamination in the presence of water is known to decrease soil darkness with increased soil diffuse reflectance (Okparanma and Mouazen, 2013b). Newer concrete has also been reported to have an overall higher reflectance than older concrete due to age

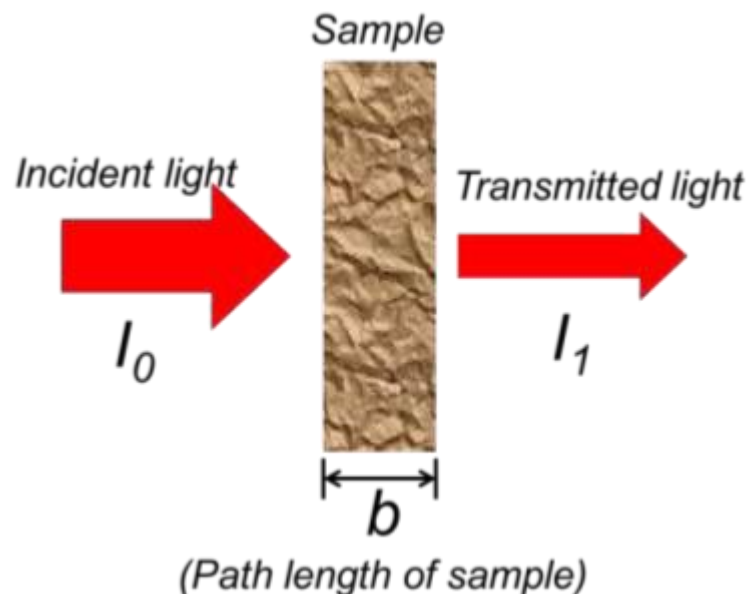


Figure 7. Process of light absorption by a sample.

and contamination with other materials such as soil and vegetation (Fairbairn, 2013). This may be explained by the

change in colour of concrete with age; the newer concrete being lighter in colour than the darker older concrete. On transparency of solid materials, though, increased transparency of newer materials results in overall decrease in reflectance while older materials with rougher surfaces have higher reflectance (Nasarudin and Shafri, 2011). From classical physics, it is known that when light is incident on solid material, some of the light is absorbed and electrons within a molecule are excited as they transit from a lower energy level to a higher one. This occurs at certain wavelengths where the energy of the incident light corresponds to an electronic transition within the molecule. The resulting absorption spectrum, which is a function of absorbance and the wavelength at which the absorption occurs, is often used to identify the chemical constituents of the material.

In the IR region of the electromagnetic spectrum (Figure 5), molecular vibration is due to absorption of light by sample linked with the type of chemical bonds between atoms in a molecule. The process of light absorption by a sample is illustrated in Figure 7. With soil sample, light absorption by a sample occurs if $I_t < I_0$ (Figure 7).

Absorptions in the NIR region require short path length of sample. Energy absorption by a sample is explained by simple harmonic (parabolic) approximation. Absorptions in the IR region are due to overtones and combinations of fundamental absorptions (occurring in the MIR spectral region) due to deviation in molecular vibration from the harmonic approximation (discussed shortly), which holds true only when almost all the molecules are in their vibrational ground state (fundamental vibration) in which the dominant spectral transition is the fundamental transition (Atkins and de Paula, 2002). This is because most absorption in the NIR region requires higher vibrational energy (Davies, 2005). At high vibrational excitation, the parabolic approximation tends to break down at some regions in the potential energy curve and the concomitant anharmonicity results in overtones and combinations of fundamental vibrations (Atkins and de Paula, 2002; Osborne et al., 1993; Pasquini, 2003). With hydrogen atom dominating NIR spectroscopy (forming C–H, O–H, N–H, and S–H chemical bonds), these overtones and combinations are assigned bands as shown in Figure 8 involving different modes of hydrogen stretching and bending. Stretching refers to vibration involving axial change in the interatomic distance between two bonding atoms while bending refers to vibration involving change in bond angle (Osborne et al., 1993). Stretching could be symmetrical or asymmetrical while bending could be scissoring, rocking, wagging, or twisting as dictated by the direction of bond vibration (Osborne et al., 1993). Since overtones and combinations are of broad bands (several wavelengths), chemometrics or machine learning techniques are commonly used to extract useful quantitative and qualitative information from the vis-NIR spectra.

Chemometrics and NIR spectroscopy

The initial set back in the study of NIR spectra was partly attributed to the requirements of a knowledge of

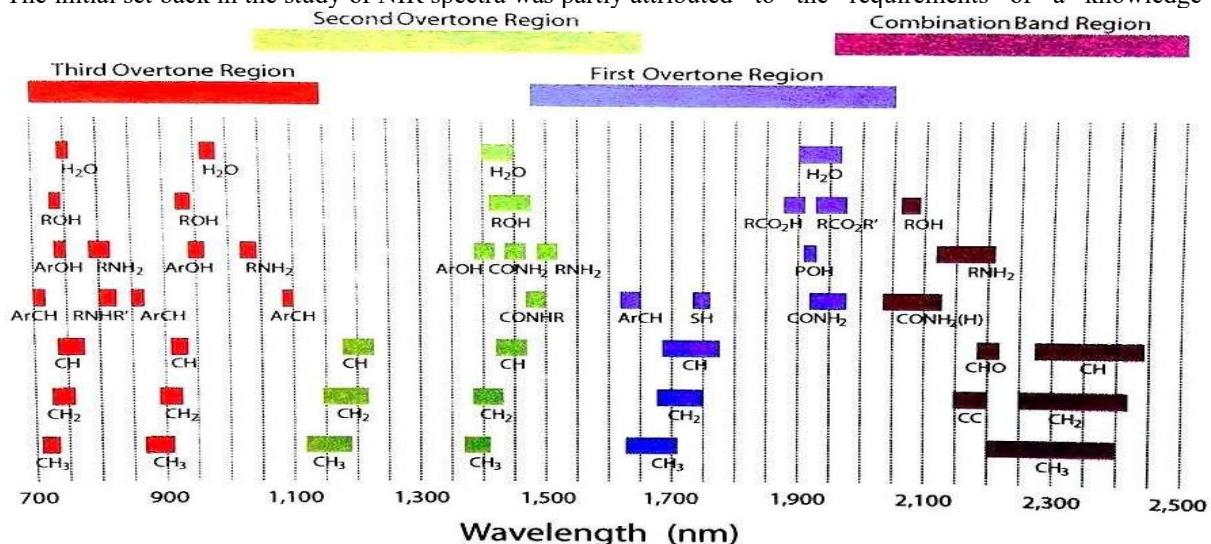


Figure 8 NIR band assignment table.

Source: Metrohm Monograph 8.108.5026EN – A guide to near-infrared spectroscopic analysis of industrial manufacturing processes. Metrohm AG, CH-9101 Herisau, Switzerland.

chemometrics, the computer, and use of very low-noise spectrometers; and the person who put all this together was Karl Norris (Davies, 2005). Chemometrics is the use of advanced statistics and mathematics in multivariate analysis (MVA) — calibration and classification. The apparent ‘miscibility’ between chemometrics and NIR spectroscopy stems from the dependence of diffuse reflectance measurement on the highly variable physical properties of a sample. This variability in physical properties causes changes in the resulting spectrum. So, to extract the relevant information necessary for a given analytical procedure, variables as high as between 20 and 1000, in most cases, need to be considered (Pasquini, 2003). This usually is implemented by employing both linear and non-linear MVA for qualitative and quantitative applications. Some linear MVA techniques that have been adopted include multiple linear regression (MLR), principal component regression (PCR),

RESEARCH NEEDS/KNOWLEDGE GAPS

In the fields of soil science, petrochemical engineering, environmental science, and civil engineering, vis-NIR diffuse reflectance spectroscopy has obviously found widespread use. Research by scholars such as Song and Saraswathy (2007), Brook and Ben-Dor (2011), Toda et al. (2012), Lee et al. (2012), and Abbas (2015) has made significant strides in filling important knowledge gaps regarding the use of vis-NIR diffuse reflectance spectroscopy to determine chloride concentration in concrete structures. However, there is still more work that needs to be done in this area. To gather more information inside the NIR circle on the determination of

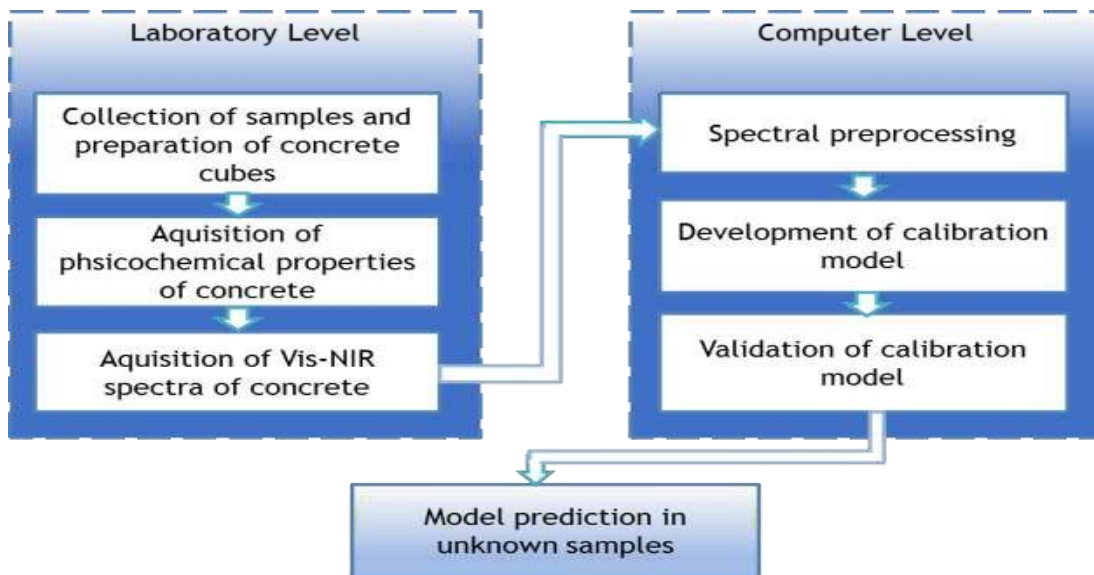


Figure 9. Proposed framework for application of vis-NIR spectroscopy in the physicochemical characterization of concrete produced with pre-treated drill cuttings.

other equally important physicochemical characteristics of concrete produced with pre-treated drill cuttings such as heavy metals, TPH, permeability, bulk density, compressive strength etc. This concrete characterization approach will significantly help to enhance the sustainable reuse of pre-treated drill cuttings as part substitute for fine aggregate in concrete by rapidly and economically characterizing resulting concrete to ensure that it complies with recommended minimum compressive strength and environmental guidelines for the protection of ecological health well into the

future.

CONCLUSION

This review has shown that drill cuttings are the solid component of the waste stream from crude oil drilling operations. They consist of hydrocarbons and heavy metals among other inorganic and organic elements; they are byproducts of the chemical composition of the customized drilling mud that is used during the drilling operation. The benthic and edaphic flora and fauna are negatively affected by the land and sea disposal of untreated drill cuttings. Numerous strategies exist for the management of drill cuts, including (among other things) its sustainable utilization for civil engineering projects; and a number of elements are coming together to make this choice more appealing. Traditional concrete characterization methods have their drawbacks, such as labor-intensive sample preparation protocols, high analytical and labor costs, and occupational health risks. As a result, researchers are increasingly focusing on vis-NIR spectroscopy as an analytical technique because it is faster, cheaper, more portable, and poses fewer risks.

There may be a chance to use vis-NIR spectroscopy to determine other important physicochemical properties of drill cuttings and concrete made with pre-treated drill cuttings, such as hydrocarbons, compressive strength, and heavy metals, based on the current reports of its use in soil science, petrochemical and environmental engineering, and the prediction of chloride in concrete.

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