Hydrocarbon-Contamination of Soils: The Potential of Sodium Clays to Decelerate Soil Toxicants

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Abstract

Hydrocarbon-contamination can change hydraulic conductivity (HC) in soils, and hence increase the spreading rate of aqueous toxicants in the ground. A constant head permeameter used in the laboratory to measure HC of soils taken from near the Pitch Lake in Trinidad determined that the HC exceeded that of a reference soil having "normal HC" for a loam. Although water moved rapidly through it, the Pitch Lake soil (PLS) remained dry due to water repellence. Treatment consisted of either of two red mud bauxite wastes mixed at 25 and 50% w/w with PLS at air dry. One of the bauxite wastes had undergone treatment with gypsum several years before and hence contained a greater proportion of calcium ions compared to the other red mud which contained more sodium ions. At 25% w/w the non-gypsum-treated red mud waste decreased HC of the PLS by 50%, and at 50% w/w caused a 10-fold decrease of HC on the PLS. The gypsum-treated red mud waste had no effect on the HC of the PLS. The drastic decrease in HC of the hydrocarbon-contaminated soil implies blocking of hydraulic channels by inorganic particles. The high levels of Na⁺ released in the Bayer beneficiation process dispersed and released fine < 5 mµ clay particles from the non-gypsum-treated red muds. This suggests that the rapid movement of aqueous pollutants in such hydrocarbon-polluted soils could be similarly curtailed under field conditions.

Keywords: hydrophobic soils, saturated hydraulic conductivity, sodicity, water repellence

1. Introduction

Water repellent soils normally exhibit low infiltration rates (Blackwell, 2000). Hydrophobicity can reduce the affinity for soils to water such that infiltration or wetting may be delayed for periods ranging from as little as a few seconds to in excess of weeks (Hall, 2009). Soil hydrophobicity is thought to be caused primarily by a coating of long-chained hydrophobic organic molecules on individual soil particles, thereby influencing soil hydrological and ecological functions (Takawira et al., 2014). A study by Lourenco et al. (2015) confirmed the hypothesis that hydrocarbon contamination induces water repellence and reduces soil moisture retention at low suction (< 100 kPa) for laboratory contaminated soils. Water is not easily absorbed by such non-wetting soils. Yet, in a pilot study of the Trinidad Pitch Lake soils at La Brea:

- (1) Water moved rapidly through the soil fabric, and;
- (2) The soil remained completely dry, despite prodigious movement of water through it;
- (3) Soil crumbs (> 2 mm), though denser than water, literally floated on water;
- (4) Soil crumbs > 2 mm exhibited mutual repellence when floated in water.

Therefore, quite apart from the last three more esoteric above-mentioned reactions, it was concluded that aqueous contaminants released in such soils, could move just as quickly. There exists therefore, the potential (Figure 1) to rapidly contaminate large volumes of soil during toxic aqueous spills.



Figure 1. A typical soil entity with no barriers to internal wetting of individual crumbs

However when crumbs are coated and sealed with hydrophobic materials water fails to enter the crumb, not wetting them. Slow wetting does not occur. For the hydrocarbon contaminated pitch-lake soil of Trinidad, water through-flow rate abnormally increases as it bypasses the soil crumbs.

Surface seepages of petroleum hydrocarbons cause hydrophobicity in soils adjacent to the Pitch Lake in southern Trinidad. In other fractured fields where surface seepages occur, there may be potentially similar results. Non-wetting soils are often prone to dispersion and structural breakdown (Ward et al., 2015). Water repellence poses challenges to stability of building foundations (Pietruszczak & Oulapour, 1999), pesticide concentration, and leaching (Blackwell, 2000), and for crop production in terms of crop establishment, nutrition, and weed control (Ward et al., 2015). The exact chemical composition of substances responsible for the development of water repellence in soils is difficult to identify, but they are generally organic compounds that accumulate on and between soil particles (Dekker et al., 2009). Two approaches to the modification of soil hydrophobicity, and hence water movements, are (1) inorganic and (2) organic.

1.1 Inorganic Approach

Water retention in soil is critical for remediation of contaminated sites (Lourenco et al., 2015). According to Roy and McGill (2002), soil water repellence is a function of soil surface chemistry. For example, abrasion of sand particles during light sieving had only small effects on repellence, but more vigorous abrasion through rotational movement of the sand reduced repellence markedly (King, 1981). Alternatively, soil hydraulic conductivity (HC) is related to the composition and concentration of exchangeable cations and soluble electrolytes (Frenkel et al., 1992). Thus Quirk and Schofield (1955) showed that the HC of a soil decreased with increasing exchangeable sodium percentage (ESP) and decreasing electrolyte concentration in the soil solution. Essington (2004) described dispersive conditions as mutual repulsion of tactoids fully surrounded by associated Na⁺ and waters of hydration (ESP < 15). McNeal et al. (1966) found a linear relationship between reduction of HC and the degree of macroscopic swelling of the extracted soil clay. Thus de-flocculation and movement of clay into the conducting pores was proposed as the second mechanism for explaining the reduction in HC. The decrease in soil HC due to swelling and dispersion of clay is explained by the presence of monovalent exchangeable cations. Such changes were responsible for the loss of nearly all drainable macro-pores in the soil, which was evidenced by the considerable reduction in cumulative infiltration amounts and rates (Lourenco et al., 2015).

Clay mineralogy has also been shown to have a large influence on reductions in HC (Churchman et al., 1993; McNeal & Coleman, 1966). Due to their 2:1 layer structure which accommodates a high amount of exchangeable Na+ within its interlayer space (Arienzo et al., 2012; Churchman et al., 1993), smectites show extensive swelling and dispersion. Na⁺ is a large monovalent ion and more effectively forces clay tactoids (*i.e.*, particles) apart than Ca²⁺ or Mg²⁺ (Quirk, 1986). Swelling occurs with increasing Na⁺ concentration, as hydration of Na⁺ leads to the expansion of the interlayer (ESP > 15).

1.2 Organic Approach

Rainfall infiltration into water repellent soils is characteristically patchy, resulting in patchy and staggered crop germination of crops and of weeds (Ward et al., 2015). They speculated that improvements in soil water

availability, and increases in organic matter, might encourage microbial activity, leading to increased degradation of the compounds causing water repellence.

Roper et al. (2013) showed that soil water contents in water repellent sand were greater in crop rows where crop residue was retained. Based on observations, Ward et al. (2015) determined that soils in crop rows showed lower levels of water repellence when crop rows were established on or close to previous crop rows, compared with crop rows established on the inter-row spaces. This was attributed to the likelihood of greater organic matter and nutrient accumulation, the former increasing water availability, which could have encouraged microbial activity (Blackwell et al., 2014). Such microbial activity could have led to increased degradation of the compounds causing water repellence (Walden, 2015). Nevertheless, compared to naturally-occurring and fire-induced hydrophobicity, limited information is available on the impacts of hydrocarbon contamination on water repellence and hydraulic properties (Lourenco et al., 2015). Increased saturated hydraulic conductivity associated with laboratory contaminated soils contradicted their original hypothesis (Lourenco et al., 2015) such that their findings imply that storms falling on initially dry recently contaminated soils may trigger contaminant transport and erosion via enhanced surface runoff, and rapid spreading of contaminants once they reach the groundwater systems.

1.3 Aim & Hypothesis

The aim of this study is to determine, for a hydrocarbon-contaminated non-wetting soil, (1) the hydraulic conductivity and hence (2) the likelihood of rapid spread of aqueous pollutants, and (3) a potential method to reduce the spreading rate of such contaminants in that hydrocarbon-contaminated soil.

Sources of Na^+ bentonite, which can reduce infiltration rates are rare in the Caribbean. Therefore it is hypothesized that abundant Na^+ -rich dried red muds from bauxite waste (of relatively low toxicity) could release reduce very high infiltration rates in hydrophobic hydrocarbon-contaminated soils.

2. Materials and Methods

2.1 Site Details

The Pitch Lake at La Brea occupies 36 ha in southwestern Trinidad at 62° W and 10.5° N (Figure 2). Average annual rainfall at La Brea is 1250-1750 mm, most of which falls between May and December. Temperature is lowest in January when it averages 25 °C, rarely rising higher than 33 °C in June through September, which are the hottest months.



Figure 2. Location of the Pitch Lake at La Brea (circular dot) in Trinidad & Tobago

2.2 Soil Sampling

Soil cores were collected on the periphery of the Pitch Lake along a transect perpendicular to the centre in April 2012, to a depth of 20 cm and brought to a laboratory on the same day. The soil was brought to air dry after exposure for three days. Black petroleum tar contaminated some soil aggregates (Figure 3) at the site. Table 1 shows that organic carbon greatly dominated this soil, at > 10 times the normally expected levels.



Figure 3. Hydrocarbon-contaminated soil on periphery of Pitch Lake in LaBrea, south-western Trinidad *Note*. The diameter of each circular indentation in the background at right is 1 mm.

Table 1a. Some properties of an undisturbed reference soil from Mount Nelson, Jamaica, and a Pitch Lake soil (Trinidad)

Property	Reference soil	Pitch Lake soil
Organic matter (%)	4.2	56.6
pH	5.3	6.9

Note. Soil samples were taken 100 m at depth 0-15 cm from the periphery of the pitch lake at LaBrea, Trinidad.

2.3 Water Repellence Determination

Water repellence was measured by counting the number of seconds required for water to be absorbed into the soil (WPDT) after the method of DeBano (1981). Fifty replicates were used, where water repellence is classified as follows: lower than 3 seconds = highly wetting (0); 3-6 seconds = wetting (1); 6-60 seconds = slightly water repellent (2); 60-600 seconds moderately water repellent (3); > 600 seconds = extremely water repellent (4). Samples were measured at air-dry were crushed and sieved to less than 2.0 mm to remove large organic matter and gravel.

2.4 Dispersion and Hydraulic Conductivity

Measurement of spontaneous dispersion and hydraulic conductivity were conducted respectively using the Modified Emerson Water Dispersion Test (Emerson, 1967). Based on the objective of potentially using the treatment as a barrier for aqueous contaminants in field soils, potentially large volumes of soil additive could be required to affect interflow under field conditions. Therefore, the constant head method used for hydraulic conductivity Klute (1983) was modified to include soil columns of thin cross-sections, *i.e.*, of 2.5 cm thickness.

2.5 Bauxite Wastes

Samples of gypsum-treated bauxite waste (O'Callaghan et al., 1998) were collected from Kirkvine Pond 6 during 2007 from two depths: 0-15 and 15-30 and stored at air-dry. Samples collected from an unmined, undisturbed bauxite soil within the same locality were used a reference soil. Bardossy (1982) characterized this reference soil as dominantly kaolinite, and freely draining. At air-dry, samples were grinded and passed through a 1.0 mm diameter sieve.

Table 1b. Some properties of bauxite wastes from Jamaica, and of a Pitch Lake soil from Trinidad

Bauxite wastes	pH	Organic carbon
Dried red mud	10.8	0.1
Gypsum-treated red mud	8.3	0.2

Note. Soil samples were taken at a depth of 0-15 cm for the gypsum-treated red mud, and 15-30cm for the dried red mud, subjacent to the gypsum-treated material.

2.6 Bauxite Waste Treatments

Two depths were selected as potential treatment additives because only the 0-15 cm layer was gypsum treated. It has been shown (Harris, 2008) that the gypsum applied to a depth of 15 cm had little or no physical effect on soil below 15 cm depth. Therefore the original chemistry of the RMW at 15-30 cm was not as altered as that of the 0-15 depth (GRMW). It was therefore postulated that the greater ESP and SAR of material from 15-30 cm may enhance displacement of inorganic ions in a hydraulic column. Removal of divalent ions from the exchange complex can contribute to reductions in aggregate stability and decreases in HC (Kopittke et al., 2006).

2.7 Red Mud Safety Concerns

Red mud can contain elevated levels of metals, and the pH of red mud characteristically ranges from 10 to 12 due to the use of caustic soda during the extraction process. Red mud is also known to contain technologically enhanced naturally occurring radioactive material (TENORM), including thorium and uranium. TENORM is naturally occurring radioactive material (NORM) that has been processed in such a manner that its concentration has increased (EPA, 2012).

However, concerns about the radioactivity in red mud waste were allayed because (Pinnock, 1991) showed that using 100% bauxite residue gave a dose equivalent to just over 2 m Sv y^{-1} and was judged to be acceptable. However, other work on Hungarian bauxites has recommended a maximum addition of 15% bauxite residue to avoid exceeding a level of 0.3 m Sv y^{-1} (Pinnock, 1991).

2.8 Statistics

Sample means compared using Tukey's Honestly Significant Difference ($P \le 0.05$) to examine differences in HC between treatments.

3. Results

3.1 Water Repellence

Water repellence of the PLS was found to be at the highest value, *i.e.*, extremely water repellent (4), compared with the reference soil, which had a value of highly wetting (0), *i.e.*, showing very rapid water absorbance. Rapid water absorbance is unusual for hydrophobic soils. Further, the fact that soil aggregates after several days in water, and even aggregates as small as < 2 mm remained dry after several hours of water infiltration showed the extremely hydrophobic nature of the Pitch Lake soils (Figure 4).

Table 2. Water repellence rating* of soil near a pitch lake, and that of bauxite wastes

Reference soil	PLS	RM	GR	
0	4	0	0	

Note. *Water repellence, measured by counting the number of seconds required for water to be absorbed into the soil after the method of DeBano (1981), is classified as follows: lower than 3 seconds = highly wetting (0); 3-6 seconds = wetting (1); 6-60 seconds = slightly water repellent (2); 60-600 seconds moderately water repellent (3); > 600 seconds = extremely water repellent (4).



Figure 4. Pitch Lake soil agglomerates after being subjected to through-flow in a water column

Note. Soil particles remained dry even after water flowed for several (> 30) minutes through the mass. The agglomerates are floating in the water, as proven by the lack of shadows under the soil particles at left. Each circular depression in background cloth is 1 mm in diameter.

3.2 Changes in Hydraulic Conductivity

There was little, if any, restriction to water-flow through the hydrocarbon contaminated samples. The hydrocarbon-contaminated soils (HCS) all exhibited high values for hydraulic conductivity, exceeding 150 cm min⁻¹ and exceeding that of the reference soil by > 35% (Table 3). This flow rate is similar to that through a fine-to-medium (< 2 mm) quartz sand.

When RMW was added at 25% of the volume of the PLS in the column, the RMW effect on the HC of the PLS was expected to be proportional to its volume (25%). Yet the 25% treatment of RMW changed PLS hydraulic conductivity, not, as was expected, by 25%, but by a far larger proportion, *i.e.*, by 400% (decrease) in HC (Table 3).

Table 3. Changes to h	ydraulic conductivity	(cm min^{-1})) of Pitch Lake	soils after	treatment with	dried red muds
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	N & C	HN25 & HC25	HN50 & HC50
Na ⁺ & Ca ⁺ -rich waste (N)	0	47	20
Ca ⁺ rich waste (C)	202	202	202

Note. Reference soil = 55 cm min⁻¹, H = hydrophobic soil, HN25 = H + 25% Na⁺ & Ca⁺-rich red mud, HC25 = hydrophobic soil + 25% Ca⁺-rich red mud, HN50 = H + 50% Na⁺ & Ca⁺ -rich red mud, HC50 = hydrophobic soil + 50% Ca⁺ dominated red mud.

Even more dramatically, RMW added at the rate of 50% to the PLS reduced HC in the column not by 50%, but by a 10-fold margin (1000%). Conversely, the GRMW in similarly added proportions of 25 and 50% had no effect on the HC of the PLS in the column. There was no observed or detected reaction. Though the reduction in porosity of the soil column was dependent on concentration of the additive, it must be noted that the particle diameters of all treatments, including the non-effective (gypsum-based) ones, were identical.

3.3 Hydrophobicity & Dispersion

Though all PLS soil samples along the transect taken across the periphery of the La Brea Pitch-Lake were water repellent, there was no observed pattern of soil hydrophobicity increasing inversely with distance from the centre of the Pitch Lake. This suggests a low threshold of hydrocarbon contamination required to produce hydrophobicity in a soil. The Modified Emerson Water Dispersion Test showed that the PLS exhibited slaking and dispersion at level 4 (the second highest level) after just 2 hours (Table 4). Contrastingly, no dispersive reaction was detected for the reference soil (Table 4), even after several weeks of contact with water. Though all the aggregates were water repellent, and, to varying degrees dispersive, those aggregates which visibly contained large amounts of high density black tar were the least dispersive, while the PLS with the lowest levels of

hydrocarbon contamination were the most dispersive.



Figure 5. Picture shows air bubbles from explosive reaction when dried Na+ dominated red mud makes contact with water, culminating in total dispersion and destruction after just five minutes in water. The clay particles so released, blocked pores in a hydrophobic Pitch Lake soil

Table 4. Modified Emerso	on Dispersion Test	levels of dried	bauxite waste and	Pitch Lake soils

	REF & REF	PLT & PHT	HN & HC
Na ⁺ rich waste (N)	0	4	4
Ca ⁺ rich waste (C)	0	4	0

Note. REF = reference soil, PLT = low-tar content pitch lake soil, PHT = high tar content pitch lake soil, $HN = Na^+ \& Ca^+$ -rich dominated red mud, $HC = Ca^+$ -rich red mud.

Key: Emerson dispersion levels: *1* Slight milkiness *2* Obvious milkiness, less than 50% of the aggregate affected *3* Obvious milkiness, greater than 50% of the aggregate affected *4* Total dispersion leaving only sand grains.

Source: Emerson Aggregate Test (1967).

4. Discussion

4.1 Infiltration

On the assumption of high levels of hydrocarbon contamination in the PLS, the inherently high level of saturated hydraulic conductivity correlating with hydrocarbon-contamination of the soil in this study is corroborated by Lourenco et al. (2015), who, in a laboratory simulated hydrocarbon contamination, induced soil water repellence. Saturated hydraulic conductivity (K_s) increased linearly with level of (hydrocarbon) contamination (p < 0.05; r2 ≈ 0.8), indicating rapid flow of water attributed to a reduction of the dielectric constant, and hence water–soil matrix interactions (Lourenco et al., 2015). A humic soil is defined as soil in which organic carbon exceeds 50%. Yet, the PLS, with 56% organic carbon (Table 1) is not a humic soil, there being no visible humus (Figure 6). Therefore, it is reasonable to conclude that hydrocarbons represent most of the organic C in PLS. On close microscopic inspection of the mineral soil, there was no humus visible. In this study, the soil with lower observable levels of hydrocarbons (black tar) exhibited greater dispersion. Hence the less dispersive samples occurred closest to the centre of the Pitch Lake PLS. This was against expectations.

In the samples closer to the centre of the Pitch Lake, dark, tarry petroleum occupied a greater proportion of the soil. Asphalt generally contains some of the heaviest and least volatile fractions of petroleum distillates. In the aquatic environment, asphalt will sink to bottom as a dark tarry substance. Petroleum distillates in order of decreasing volatility include:

- 1. Petroleum ether or benzene
- 2. Gasoline
- 3. Naphtha

- 4. Mineral spirits
- 5. Kerosene
- 6. Fuel oil
- 7. Lubricating oils
- 8. Paraffin wax
- 9. Asphalt or tar

As gravitational forces act on all objects including those with horizontal movement, lighter, more mobile short-molecular chain hydrocarbon fractions would have been expected to have travelled furthest out from the centre of the Pitch Lake. It is plausible then, that the smaller hydrocarbon chains of the lighter fractions would have more easily penetrated the micro-pores and microstructure of those soil aggregates in direct variation with distance from the centre of the Pitch Lake. Thus, the greater the distance, the greater the sorting and concentration of contaminants in such soil samples. This leads thereby to (1) greater effective hydrophobicity and/or (2) greater dispersion amongst such (more distant) samples.

The results of this study show that a dispersive clay synergistically decreased hydraulic conductivity in a hydrophobic hydrocarbon-contaminated soil. This outcome supports the hypothesis of this study which was based on the activity of sodium-affected soils in an aqueous medium. Even as little as 1% of the total clay when dispersed, affects the hydraulic conductivity by blocking micro-channels in the soil mass (Goldberg & Glaubig, 1987). Tuffour et al. (2015) found that finer sediments were highly effective in altering soil properties even at low concentrations. They showed that after mixing with 10% dry Na⁺ bentonite, wetting resulted in complete filling of skeletal pore space by a jell-like clay fabric. With a 3% level of sodium oxides in Jamaican red muds, the Na⁺ levels of the RMW would have been sufficiently strong to power clay dislocation, and it is dislocated clay-sized particles that changed the HC in the Pitch Lake soils of this study.

The difference between the dried gypsum-treated red mud waste at 0-15 cm depth and that of the 15-30 cm depth was still large even after 10 years. For example, it can be seen that an explosive reaction (air bubbles observed) occurred when the Na^+ -rich red muds were placed in water (hydrated, Figure 5), thus blocking pores in the PLS.

In contrast, the dried gypsum-treated red mud retained its integrity indefinitely (Figure 6) and thus had no effect on the hydraulic conductivity of the Pitch Lake soil. These samples had been in water not for 5 minutes but for five weeks.

These results suggest that the RMW released components which blocked hydraulic channels in the PLS. As described in previous studies, the negative effects of Na^+ are most pronounced in soils which are high in clay, especially high charge-density clays (Frenkel et al., 1978; Beulow et al., 2015). In the current study, the water-repellent soils were more responsive to soil with higher Na^+ quantities, than the less reactive gypsum-treated soil. Though the original bauxite soils are mainly kaolinite, red mud wastes contain an increased number of fine clay particles compared to that of the original soil.



Figure 6. Dried gypsum-treated red mud waste (GRMW) had no observable reaction with water, retaining its stability even after many weeks. Scale is supplied by 1-mm-diameter indented circles in the backgound

These provide extra negatively charged sites for adsorbing hydrated Na⁺ cations. Similarly, the tendency of Na⁺ ions to facilitate the formation of multiple layer hydrates in smectite clays leads to greater swelling and reduction of HC. On the contrary, Frenkel et al. (1992) showed that smectites did not release clay particles when added to a sandy soil unless anions were added, because the HC of smectite clay-sand mixtures decreased only following the addition of the various anions. Dispersed clay appeared in their effluent only upon addition of citrate or hexametaphosphate. Therefore, at 56% intrinsic organic matter (Table 1), such hydrocarbon contamination would have greatly increased the negative charges of the Pitch Lake soil. This could have increased repelling forces amongst the thoroughly mixed-in clay particles, thereby blocking hydraulic channels.

Further, this result conforms with the very high sensitivity of illite to even small amounts of exchangeable Na (Oster et al., 1980). They found that, for a given ESP, the critical flocculation concentrations (CFC) of illite was much larger than for smectite, and hypothesized that the explanation lies in the irregular nature of illite particles, which prevent good contact between edges and planar surfaces, thereby decreasing the potential for inter-particle attraction. Similarly, the red mud particles, having undergone beneficiation (extreme comminution), would have been altered to more irregular shapes. Compared with Sintering red mud, the Bayer Process (used in the Caribbean alumina processing) produces a relatively small particle diameter. The particle diameter of Bayer red mud is between 0.8 μ m and 50 μ m with an average value of 14.8 μ m (Wang & Liu, 2012). In the case of the smectite cited by Frenkel et al. (1992), HC is decreased through partial blocking of pores by short distance migration of dispersed particles, provided the particle size of the sand (or host soil) is sufficiently fine to retain these particles. Being a clay-loam, the particle size of the PLS is smaller than that of fine sand and therefore would be susceptible to pore blockage by the fine clay particles released from the dried red mud.

5. Conclusion

By repelling water from pore channel walls, hydrophobicity can markedly reduce resistance to movements of aqueous liquids through soils. Finely crushing RMW from < 2 mm to < 1 mm produced an at least 4-fold increase in the effective surface area of the RMW. Soil water repellence is a function of soil surface chemistry. More specifically, it is a function of the free energy of the solid/gas interface in soil (γ SG). In contrast to the low Na⁺ RMW, the high Na⁺ RMW produced dislocated clay particles which blocked hydraulic channels.

With a high energy surface exhibited by rapidly decreasing the initial advancing contact angle (θ), the high Na⁺ RMW either expanded, or released clay particles, either of which could have blocked hydraulic channels in the PLS.

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