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SELECTED ADSORBENT MATERIALS FOR OIL-SPILL CLEANUP
A thermoanalytical study

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Thermogravimetric analysis and differential scanning calorimetry have been applied to determine the adsorption of oil on selected adsorbates: sand, organo-clay and raw cotton. Thermal analysis provides evidence for the interaction and physical adsorption of the diesel oil on the adsorbates. Sand adsorbed diesel to around 33% by mass through weak physical interactions and appeared to fractionate the diesel components. The organo-clays more strongly adsorbed the diesel as evidenced by higher thermal decomposition temperatures. Differential scanning calorimetry (DSC) shows a strong interaction between the organo-clay and the diesel oil. Diesel is effectively adsorbed on organo-clay through adsorption and partitioning and is not removed from the organo-clay until significantly higher temperatures. Cotton displayed a very high adsorption/absorption capacity. A shift in the peak at 110°C (compared with pure diesel at 90°C) suggests an interaction between the diesel compounds and the cotton fibres as diesel is being retained at higher temperatures and more energy is required to evaporate the diesel. DSC was used to determine the strength of the diesel adsorption on the sand, organo-clay and cotton. The use of adsorbent materials to adsorb oil from oil spills is of great significance in modern society. One method of proving the effectiveness of an adsorbent material is through thermoanalytical techniques.

Keywords: *adsorption, cotton, DSC, hydrocarbons, organo-clays, sorbents, TG*

Introduction

Oil and petroleum products are some of the most important energy sources in the world, and as long as oil is prospected, transported, stored and used there will be a risk of spillages resulting in potentially significant environmental damage and economic loss. During land-based transport, oils and other oil-based products are spilled on roads with adverse impact on road usage, quality and repair costs as well as affecting the environment and the community. When spills occur, human health and environmental quality are put at risk. Therefore, every effort must be made to prevent hydrocarbon spills and to clean them up promptly once they occur.

Collected records between 2001 and 2005 from the relevant road maintenance authorities and emergency services agencies in the United States, Canada and Australia report hundreds of major and minor oil spillages commonly involving gasoline and diesel discharges [1–4]. Major impacts include traffic disruption to the community, road pavement degradation and significant environmental clean-up costs which can amount to many millions of dollars as illustrated by a relatively minor Australian incident (1999) when 80000 L of brake fluid were released on to a four-lane highway. This resulted in total cost of about US $2 million [5] for clean-up and environmental remediation of the surrounding soil and groundwater environment.

In general, regulations at all levels of government world-wide are becoming more strict regarding any environmental chemical spills and effective spill management regimes are required. This implies a need for sorbent materials that are effective for use in oil spill clean-up operations.

There are many sorbents in use [6] including sand as a common example, but others have been suggested as improved materials such as organo-clays and cotton fibres. Clearly all sorbents interact with the spilt oil, and it is important to understand at least some of the fundamental properties of the sorbent–sorbate interaction. Thermal analysis is a technique, which can be used to study such phenomena for all types of materials [7–15], and indeed their mixtures with the oil sorbates. In this work, thermogravimetric and differential thermogravimetric analysis, in combination with differential scanning calorimetry, has been applied to investigate some properties of selected adsorbent materials including sand, organo-clay [13, 16–23] and raw cotton, and their interactions with a typical oil adsorbate, diesel oil.

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Experimental

Materials and methods

Organo-clays

The montmorillonite used in this study was supplied by the Clay Minerals Society as source clay SWy-2-Na-Montmorillonite (SWy-2-MMT) (Wyoming). This clay originates from the Newcastle formation, (Cretaceous), County of Crook, State of Wyoming, USA. The cation exchange capacity (CEC) is 76.4 meq per 100 g (according to the specification of its producer).

The surfactant used in the modification of the clay was dodecyldimethylammonium bromide (DDDMA), $C_{22}H_{48}BrN$, MW: 406.53. The surfactant was sourced from Sigma-Aldrich.

The syntheses of surfactant–clay hybrids were undertaken by the following procedure: SWy-2-MMT was first dispersed in deionised water (the ratio of clay to water was 100 g:2.750 mL) and then stirred with a mechanical stirrer for about 10 h. A pre-dissolved stoichiometric amount of surfactant was slowly added to the clay suspension. The reaction mixtures were stirred for about 24 h. All organo-clay products were washed three times with deionised water to remove excess surfactants, filtered and dried at room temperature or in an oven at 50–60°C, ground in an agate mortar, and then stored in a vacuum desiccator for about 7 days.

Cotton

Raw cotton fibres used in the study were sourced from Queensland Cotton (QC), Brisbane. The cotton fibres were collected in the 2003 season from the following growing regions: Moura, Theodore, Dalby and Goondiwindi in Queensland, and Wee Waa in NSW. The ginned cotton fibres (lint) were a by-product from the classing process. A mix of fibre variety and types was used in the study sorbents.

Characterisation of cotton samples was not carried out since the by-product cotton bales were made up of samples from different growing regions (i.e. heterogenous sample). Therefore, the cotton samples used had a variety of cotton length, uniformity, strength, micronaire and colour. In addition, no cotton fibre ‘conditioning’ was undertaken in the experimental work. The cotton fibres were essentially used in the raw state.

Sand

Fine sand was sourced from a local supplier in Brisbane, Queensland.

Diesel

Diesel used in this study was sourced from BP Australia. Typically, BP diesel has a viscosity of 1.2–4.6 cSt @ 38°C and specific gravity of 0.84.

Thermal analysis

Thermogravimetric analysis (TG) of the samples was obtained using a Q500 TA® instruments Inc. Q500 high resolution TG operating at a heating rate 2°C min^{-1} and resolution 6.0°C from room temperature to 1000°C in a high purity flowing nitrogen atmosphere (80 mL min^{-1}). Approximately 20 mg of cotton, 25 mg of SWy-2-MMT or 95 mg sand or organo-clay was heated in an open platinum crucible. No sample preparation was required other than adding 50 mg of diesel to produce a diesel–saturated sample for analysis.

Differential scanning calorimeter (DSC) analyses were conducted using Q10 TA® instruments Inc. with a heating rate of 2°C min^{-1} from room temperature to 400°C in nitrogen (50 mL min^{-1}). Approximately 5 mg of cotton, 5 mg of SWy-2-MMT or 10 mg of sand or organo-clay was heated in a hermetically sealed aluminium pan. Again, no sample preparation was required other than adding 10 mg of diesel to produce a diesel–saturated sample for analysis.

Results and discussion

The characterisation of sorbent materials (sand, organo-clays and cotton) is problematic because the structure of the materials is enormously complex. A complete characterisation is not possible. However, average properties (or simple property relationships) can be used to provide an understanding of the sorbent materials and their sorption capacity.

Thermal analysis

Thermal analysis techniques are often used for the characterisation of materials [24–28]. The two methods used in this work include thermogravimetry (TG) and differential scanning calorimetry (DSC). TG analysis provides a method for the determination of mass change in the material as a function of time and temperature. To enhance the TG analysis, the derivative-thermogravimetric (DTG) is also used to give the onset mass loss and peak maximum. DSC allows a rapid detection and measurement of the physico-chemical changes that a material undergoes when subjected to heating. The DSC typically measures the energy evolved or absorbed by the material during analysis. Therefore, these techniques reflect reactions which occur at the molecular level of the selected sorbent materials.

Diesel has been selected as the representative hydrocarbon for thermal analysis. A brief discussion is given on the properties and thermal degradation of diesel. This information provides the basis for the assessment of adsorption capacity of the sorbent materials. By comparing the behaviour of free diesel with diesel saturated sorbents, the adsorption characteristics of the materials can be determined. Three sorbent materials were used in this analysis: sand, organoclays (SD1) and cotton fibre.

Diesel

BP automotive diesel fuel is a complex mixture of middle distillate hydrocarbons with carbon numbers in C_{10} to C_{28} range, boiling point between 180–380°C, and viscosity of 2.1–5.5 $mm^2 s^{-1}$ @ 40°C. Because of its complexity, a complete chemical breakdown of diesel is not feasible. However, the elemental and compositional data based on chemical structure are provided in Table 1.

Based on the analysis in Table 1, diesel contains four kinds of carbons in: a) straight-chain branches, b) double or triple bonds; and c) aromatic rings. The diesel mixture is particularly rich in alkanes (83.7% by mass) with average carbon number of $C_{14.2}$ while the aromatics account for the remaining 16.3% by mass. Generally, diesel molecules of different lengths have different properties and behaviours. As the chains get longer, they get heavier. The different chain lengths of diesel compounds have progressively higher boiling points.

The TG curve for free diesel showed a rapid mass loss (98%) from 30 to 180°C (Fig. 1). In the DTG curve (plotted as dm/dT) for diesel, the onset mass loss (30°C) and peak maximum (90°C) are also shown. At least four diesel fractions having different chemical composition were observed in the DTG curve where the shoulders (i.e. 55, 75, 120 and 125°C) correspond to the loss of different diesel fractions which range from C_{10} to C_{22}. A small mass loss (~0.5%) in the 180–200°C region is attributed to the heaviest diesel fractions ($>C_{22}$). It can be noted that diesel is completely evaporated at a temperature of around 200°C.

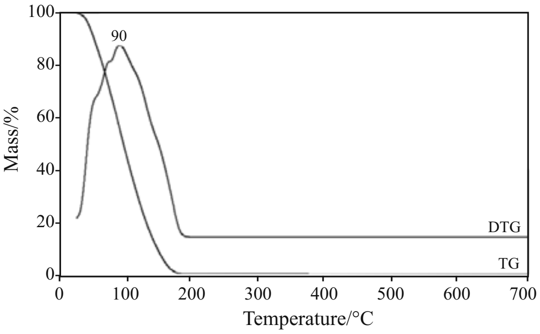

Fig. 1 TG and DTG curves of diesel showing peak maximum at 90°C and complete evaporation at approximately 200°C

Table 1 Typical composition of diesel [29]

Compound type	Mass/%	Average or reference compound	Average molecular mass	C/mass%	H/mass%
Saturates	**83.7**	$\mathbf{C_{14.2}H_{29.4}}$	199.6	0.853	0.147
Paraffins	38.7	$C_{16}H_{34}$	226	0.850	0.150
Cycloparaffins	29.6	$C_{10}H_{21}$	141	0.851	0.149
2-ring cycloparaffins	11.5	$C_{16}H_{32}$	224	0.857	0.143
3-ring cycloparaffins	4.0	$C_{22}H_{38}$	302	0.874	0.126
Aromatics	**16.3**	$\mathbf{C_{9.5}H_{11}}$	125.6	0.912	0.088
Mono-aromatics	10.5	$C_{9.2}H_{10.4}$	121.1	0.914	0.086
Benezenes	7.3	C_8H_8	104	0.923	0.077
Naphthene benzenes	3.2	$C_{12}H_{16}$	160	0.900	0.100
Dinaphthene benzenes	0	NA	NA	NA	NA
Di-aromatics	5.7	$C_{10.3}H_{12.3}$	136.1	0.910	0.090
Naphthalenes	1.8	$C_{13}H_{14}$	170	0.918	0.082
Acenaphthenes	3.5	C_9H_{12}	120	0.900	0.100
Fluorenes	0.3	$C_{13}H_{10}$	166	0.940	0.060
Tri-aromatics	0	NA	NA	NA	NA
Tetra-aromatics	0	NA	NA	NA	NA
Total	**100**	$\mathbf{C_{13.4}H_{26.3}}$	**187.5**	**0.857**	**0.141**

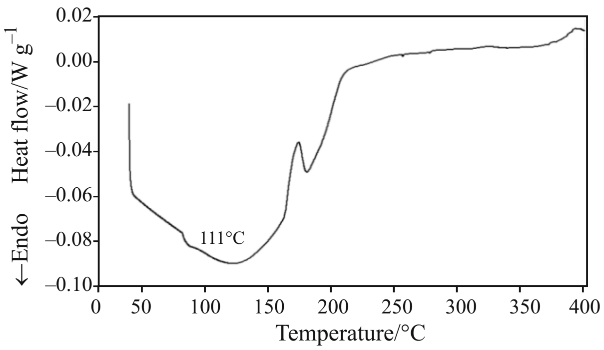

Fig. 2 DSC curve of diesel showing broad endothermic peaks (shoulders) at 25 and 200°C and peak temperature at 111°C

The total mass loss observed in the TG curve corresponds to the evaporation of diesel in the DSC curve which gave rise to broad endothermic peaks (or shoulders) at 25 to 200°C, with a peak temperature at 111°C (Fig. 2). The broad endothermic peaks are indicative of the complex mixture of diesel (i.e. C_{10} to C_{28}). The lighter diesel fractions (volatiles $<C_{12}$) tend to evaporate at the lower temperature end and the heavier diesel fractions ($>C_{16}$) tend to evaporate at the higher temperature end [26]. The peak temperature (111°C) possibly corresponds to the average carbon fraction ($C_{14.2}$) of the mixture.

Sand

Although not recognised as a sorbent material, sand has been widely used by Queensland Department of Main Roads for hydrocarbon spills on roads. This is mainly due to its availability and low cost. Therefore, sand has been adopted as a reference material in this study.

TG and DTG curves for sand and diesel–saturated sand are shown in Fig. 3. There are no mass losses associated with sand in this temperature range because sand melts at extremely high temperatures (i.e. >1790°C). However, sand does undergo a transformation (phase change) from low temperature quartz phase to high temperature quartz phase (at 570°C), resulting in a different crystalline structure and properties [30]. For diesel–saturated sand, a mass loss of 33% was observed in the TG curve from 25 to 200°C. This mass loss was attributed to the evaporation of diesel adsorbed on the sand particles. This may mean that sand adsorbs 33% of diesel by mass. In the DTG curve, there was a shift in the peak maximum of 90°C (pure diesel) to 108°C (sand saturated with diesel). This result suggests that diesel is being retained/adsorbed at significantly higher temperatures by the sand. The diesel fractions may be redistributed, with lighter components being retained at higher temperatures relative to the heavier fractions. This may account for the more rounded shape of the DTG curve for diesel–saturated sand sample – diesel is evaporating at a consistent rate.

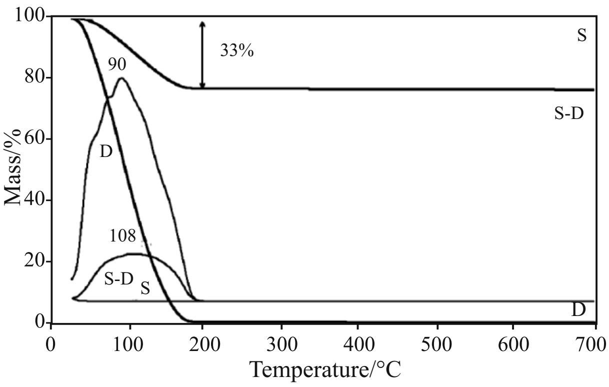

Fig. 3 TG and DTG curves for diesel (D), sand (S) and sand saturated with diesel (S–D)

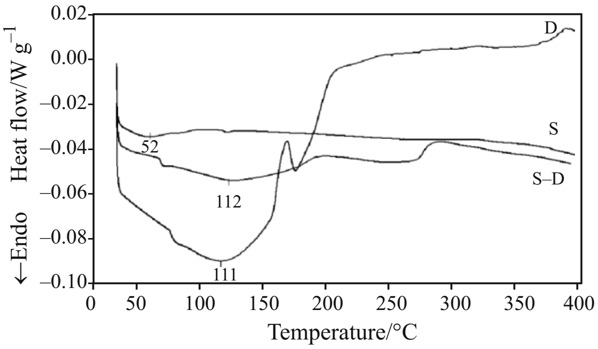

Fig. 4 DSC curves for diesel (D), sand (S) and sand saturated with diesel (S–D)

In the DSC curve, the endothermic peak at 52°C is related to the loss of moisture adsorbed on sand particles (Fig. 4). This loss is relatively small, as designated by the shallow endothermic peak. In terms of uptake of diesel by sand, the endothermic peak temperature for sand and diesel–saturated sand is similar (~111°C), which suggests that diesel is only weakly retained by sand. This may be due to the sand substrate which is not conducive to interaction with diesel since it is non-porous and relatively hydrophilic. Thus, diesel is only adhering to the sand particles due to weak dispersion forces.

Organo-clays

Thermal analysis techniques have been widely used to study organo-clays and their structure [24, 28, 31, 32]. In this study, the focus is on: a) the assessment of thermal reactions of organo-clay (SD1); and b) investigation of the effect of the organic materials (surfactants) on the amount and properties of adsorbed diesel. Yariv [28] defines the following thermal processes for organo-clays: a) dehydration of adsorbed water (<200°C); b) region of thermal reaction of organic material (200–500°C); and c) dehydroxylation (i.e. evolution of water from hydroxyls belonging to the clay layers) and transformation of the clay into crystalline phases (>500°C). Xi *et al.* and He *et al.* have reported the thermal decomposition of organoclays [13, 20, 21, 33]. These workers showed that the adsorption of organic molecules depended on the molecular arrangement of the surfactant molecules in the clay interlayer. It is anticipated that these organoclay thermal decomposition features will be observed in the TG and DTG curves obtained in the study. Below 200°C the surfactant starts to degrade and evacuation of head amine groups occurs. Simultaneously unsaturated hydrocarbon didecene is formed. This component may be assumed as additional component of diesel. Desorption from the surface, evacuation of bulk components from pores and surfactant degradation take place in the same range of temperature and probably all these effects overlap. Thus, interpretation of TG, DTG and DSC curves is rather difficult.

In the TG and DTG curves of the organo-clay (SD1), there is a small mass loss (~3%) attributed to the dehydration (>200°C) (Fig. 5). However, the area of most interest in the TG curve is the three main mass loss regions in the 180–400°C temperature range (i.e. 202, 270 and 370°C). The mass loss in this region is related to the loss of the organic surfactants in the clay layers [28, 31]. He *et al.* [14] observed three peaks (ca. 210, 280 and 375°C) in the DTG curve of hexadecyltrimethylammonium bromide (HDTMAB) modified montmorillonite. These three mass loss peaks were attributed to the evaporation/decomposition of the loaded surfactant. The peak at 210°C was related to the evaporation of surface surfactants (outside the clay layer) while the other two (280 and 375°C) corresponded to the decomposition of the intercalated surfactants within the clay interlayer.

Thus, the three observed DTG peaks in Fig. 5 correspond to [31]: a) first peak at 202°C is due to the evaporation of surfactants on the clay surface; b) second peak at 270°C is attributed to the decomposition of surfactants adsorbed in the clay interlayer (i.e. intercalated through physical adsorption) and c) third peak at 370°C is attributed to the decomposition of the surfactants intercalated through ion exchange. The mass loss corresponding to the DTG peaks total 8% for surfactants on the clay surface and 14% for surfactants in the clay interlayer (Fig. 5). As the primary adsorption mechanism is between the diesel compounds and the hydrophobic alkyl group on SD1, the location and amount of surfactants on the clay are very important. In the majority of cases, the surfactant is both physically adsorbed to the clay surface and intercalated between the clay layers [31]. This suggests that the adsorption mechanism may involve surface adsorption and partition.

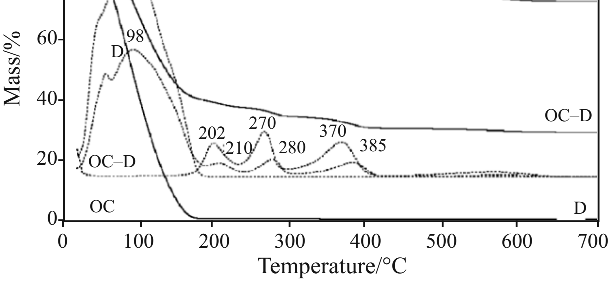

Fig. 5 TG and DTG analysis for diesel (D), organo-clay (SD1), organo-clays saturated with diesel (SD1–D)

By comparing the DTG curves for pure diesel and diesel–saturated SD1, the following observations were made:

- Diesel degradation region (<200°C): DTG peak for diesel has shifted from 90°C (pure diesel) to 98°C (diesel–saturated organo-clay). This means that the diesel fractions are being retained at higher temperatures. The BET results for SD1 suggest that surface adsorption is primarily responsible for the uptake of diesel [34]. In addition, the presence of surfactants on the clay surface and interparticle pores act to form an organic medium for the adsorption of diesel molecules through a partition process [35].
- Surfactant decomposition region (200–425°C): DTG peaks for the evaporation of surfactants has shifted to higher temperatures as shown in Fig. 5. This is likely to be due to the influence of diesel on the organic surfactants. There are two possible ways that diesel can interact with the surfactants to cause a shift in the peaks. The first method may involve diesel residue coating the organo-clay and preventing the degradation of the surfactants. This may be the case if the partition process is occurring between the diesel compounds and the organo-clay (i.e. retention of higher molecular mass component relative to the evaporating lighter fractions). The second method may involve re-ordering of the surfactants due to interaction with diesel. As the surfactants are more ordered in the clay interlayers, the degradation process is occurring at higher temperatures [26, 36, 37]. The desorption of diesel from the organo-clay depends upon the clay surface and the clay pores. If the capillaries are narrow then higher temperatures are required to desorb the diesel.

A comparison between the diesel and organo-clay (SD1) DSC curves was also carried out (Fig. 6). The DSC curve for SD1 showed two broad endothermic peaks: a) first temperature range (30 to 125°C),

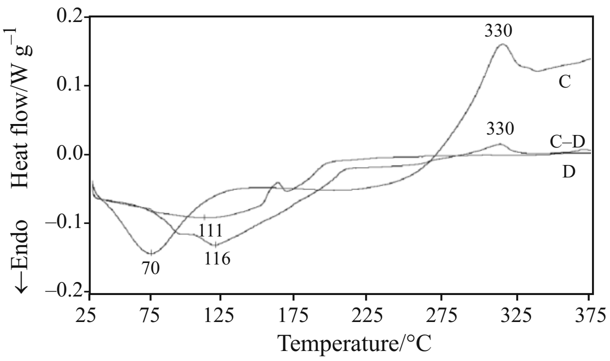

Fig. 6 DSC analysis for diesel (D), organo-clay (SD1) and organo-clay saturated with diesel (SD1–D)

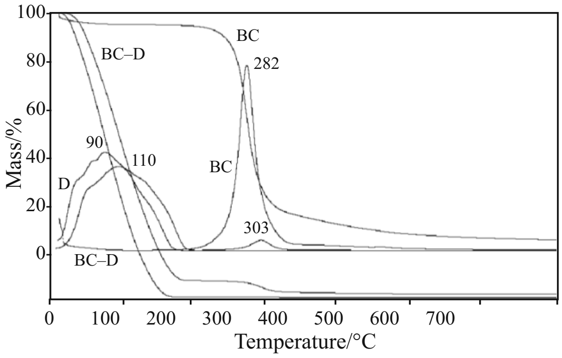

Fig. 7 TG and DTG analysis for diesel (D), cotton fibre (BC), cotton fibre saturated with diesel (BC–D)

with a peak temperature of 81°C, was attributed to the loss of free and interlayer water; and b) second temperature range (200 to 375°C), with a peak temperature at 255°C, was attributed to the evaporation of the surfactants. These observations were also noted in the TG/DTG curves (Fig. 5). In terms of diesel–saturated SD1, broad endothermic peaks (or shoulders) at 25 and 200°C, with a peak temperature at 116°C, corresponded to the loss of free water and weakly held diesel. Information such as whether or not the surfactants interact with the diesel and the strength of that interaction may be obtained from the evolution (peak) temperatures in Fig. 6. The shift in peak temperature from 111 to 116°C, indicates that uptake of diesel loosely bound (adsorbed) by the organo-clay is moderately strong compared to sand. Adsorbed diesel is likely to bind to the surfactants on the clay (surface) layers and within the interparticle pores (i.e. surface adsorption as well as partition). The retention of diesel by SD1 at higher temperatures is illustrated in Fig. 6 where SD1–D showed endothermic peaks at 25–225°C, compared to pure diesel endothermic peaks at 25–180°C. In addition, the broad endothermic peak for SD1–D implies that more energy is required to vaporise the heavier diesel compounds. Therefore, the DSC analysis does illustrate that there is some interaction between the organic surfactants and the diesel compounds.

Cotton fibre

Thermal analysis techniques have played important roles in understanding the thermal degradation of cotton fibre [25, 27, 38, 39]. Thermal degradation of cotton involves three main (overlapping) chemical reactions [40]:

- decomposition of the glycosyl units of cellulose, accompanied by evolution of water, carbon dioxide and carbon monoxide, to form a charred residue (<300°C);
- depolymerisation of the cellulose macromolecule to form levoglucosan, accompanied by decomposition of the original polymer and anhydro-sugar products (300–600°C); and
- direct fission of the substrate and the intermediate products to form lower molecular mass gaseous products (>600°C).

To provide an understanding of the uptake of diesel by the cotton fibres, the thermal degradation of diesel–saturated cotton fibres was compared with the thermal degradation of cotton fibre and free diesel.

The TG and DTG curves for diesel (D), cotton fibre (BC) and diesel–saturated cotton (BC–D) are shown in Fig. 7. There are three main mass loss regions in the TG curve for the BC cotton fibre. The initial mass loss of about 7% at <200°C is related to the evaporation of adsorbed water. The second at 270–375°C is attributed to the decomposition of the cotton fibres. The mass loss associated with the decomposition of cotton in this region is about 66%. The third region is the gradual mass loss associated with the char residue (>375°C). In addition, the DTG curve reveals that the peak maximum for BC cotton fibre occurs at 282°C.

In comparing BC and BC–D samples, the major difference is observed in the region below 200°C. The rapid mass loss below 200°C is attributed to the evaporation of pure diesel and adsorbed water. As cotton fibres are lighter than diesel, the mass loss associated with diesel clearly dominates in the TG curve. However, the main feature associated with the decomposition of cotton is still evident in the temperature range 250–375°C with the peak at 303°C. The mass loss for diesel and cotton is about 96 and 4%, respectively.

As stated, this region is of most interest since it contains the most information about diesel and its interaction with BC cotton fibre. In the DTG curve, a shift in the peak for BC–D at 110°C compared with pure diesel at 90°C was observed. This suggests that there is an interaction between the diesel compounds and the cotton fibres as diesel is being retained at

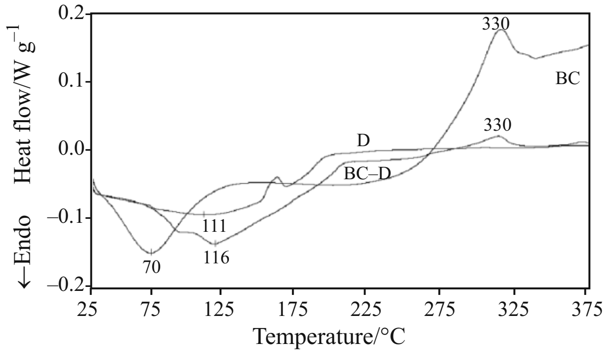

Fig. 8 DSC analysis for diesel (D), cotton fibre (BC), cotton fibre saturated with diesel (BC–D)

higher temperatures and more energy is required to evaporate the diesel.

In the DSC curves, BC cotton showed two broad peaks:

- first is the endothermic peak at about 70°C and is attributed to the evaporation of adsorbed water and
- second is the exothermic peak at about 330°C which corresponds to the decomposition of BC cotton fibre (Fig. 8).

The shift in the endothermic peak temperature from 110°C for pure diesel to 116°C for BC–D confirms that more energy is required to evaporate the diesel off the cotton sample. These observations were also displayed in the TG and DTG curves.

The BET analysis for BC cotton indicates that the adsorption process predominately occurs at the fibre surface since the internal pores were inaccessible to N_2 adsorption [34]. This was a function of both the sample preparation method (i.e. drying under vacuum at 120°C) and the dry state of the original fibres. However, the BET surface area for BC (8.2 $m^2\ g^{-1}$) suggests that there were only limited sorption sites on the surface of the fibre [34]. Therefore, other adsorption mechanisms must be responsible for the high sorption capacity of cotton cited in literature (>40 $g\ g^{-1}$) [41].

One possible adsorption mechanism is interaction between the cotton wax, located in the primary wall of the fibre, and the diesel compounds. The wax is a mixture of high molecular mass, long chain, mainly saturated fatty acids (with even numbers of carbon atoms, C_{28}–C_{34}) and alcohols (with even numbers of carbon atoms, C_{28}–C_{34}), resins, saturated and unsaturated hydrocarbons, sterols, and sterol glucosides [42]. The wax layer (~1 mass%) is responsible for the hydrophobic property of raw cotton fibre. Therefore, cotton wax may facilitate the uptake of diesel compounds through non-polar interaction. In addition to the wax layer, the morphology of the cotton fibre may also facilitate uptake of diesel compounds through the use of interfibre capillaries. This premise was supported by the results of an ESEM study [34].

Conclusions

The main adsorption process of the sorbents under investigation was surface adsorption in the diffusion region through weak dispersion forces (sand and SD1 organo-clay) and uptake by interfibre capillaries (BC cotton fibre). Thermoanalytical tests were used to characterise and assess the adsorbent performance. Sand adsorbed diesel to around 33% by mass through weak physical interactions and appeared to fractionate the diesel components. The organo-clays more strongly adsorbed the diesel as evidenced by higher thermal decomposition temperatures. DSC shows a strong interaction between the organo-clay and the diesel oil. A shift in the peak at 110°C (compared with pure diesel at 90°C) suggests an interaction between the diesel compounds and the cotton fibres as diesel is being retained at higher temperatures and more energy is required to evaporate the diesel.

It is concluded that diesel is adsorbed more strongly on cotton and organo-clays compared with adsorption on sand. This leads to the conclusion that both raw cotton and organo-clays are suitable for the adsorption of diesel oil and hence have the potential for use in oil-clean-up situations.

Acknowledgements

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