Potentially Exploitable Base-Metal Containing Bentonite Clay Minerals of Ibeshi-Ikorodu South-Western Nigeria for Oil Bleaching

By

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***Abstract*:** The experiment has been undertaken to evaluate the bleaching capacity of Ibeshi Montmorillonite-Bentonite clay to be used on soya bean oils (edible oils). The clay capacity for bleaching was evaluated before and after acid activation processes. The acid-activated Bentonite was prepared from raw Bentonite with sulphuric acid of concentrations 6 M, 7 M, and 8 M; this is because availability of hydrogen is a competitor at ions exchange site. Acid activation promotes catalytic activities by increasing the number of active sites of the clay samples. The results from AAS, GC-MS analysis and the spectra of the raw clay indicated that the dominant components present were Al_2O_3, MgO, CaO, Na_2O, and K_2O together with Fe_2O_3, TiO_2, MnO_2 and P_2O_5. The ratio of Na_2O:CaO is 0.24-0.30, a value less than one, indicative the presence of montmorillonite, and the $SiO_2 : Al_2O_3$ between ratio of 1.12-1.50 greater than one. The optimum acid concentration for industrial bleaching is 6 M H_2SO_4. However, the exchange capacity was observed at pH 7. The work has shown that activated montmorillonite/Bentonite clay has other useful organic compounds such as organic complexes 2, 4-Nonadienal; up to 8% α-Tocopherol. Tests for bleaching performance evaluation confirmed that the clay has moderately bleaching action as shown by percentage colour reduction. The colour reduction for natural clay was 9.1%, this value increases to 27.3% after 8M H_2SO_4 activation. Other properties include the structural characteristics, free fatty acid value, viscosity were also recorded. The availability of

Bentonite across Nigeria makes it a potential industrial mineral for the economy development.

Keyword: Bentonite, clay, dominant, components, acid-activated

Introduction

Montmorillonite (Bentonite) belongs to smectite group, monoclinic silicates of general formula $X_{0.3}Y_{2-3}Z_4O_{10}. nH_2O$, here X is exchangeable ions = Ca/2, Li, Na; Y = Al, Cr^{+3}, Fe^{+2}, Fe^{+3}, Li, Mg, Ni, Zn; Z = Al, Si. Bentonite is formed after volcanic ash has weathered and aged in the presence of water. It has a strong negative electromagnetic charge. Bentonite clay is a very unique substance, formed primarily of Montmorillonite, which is an extremely flat crystal flake that carries a relatively strong negative ionic charge [1-2]. The negative charge is compensated for by adsorbing a cation (sodium, calcium, potassium, sodium) to the interior of the molecule, (Figure 1); this is what makes it base-metal containing Bentonite clay mineral. The clay can be acid activated. The Montmorillonite, a three layered type of clay has octahedral sheet between two tetrahedral sheets. The ore consists essentially of aluminum silicate sheet structures, and the exchangeable cation is located in interlayer positions or adjacent to the particular surface. When particles of aluminum silicates are placed in the aqueous/polar solution, the interlayer and surface cations are displaced and replaced quickly, by cations from solution and the mineral grains subsequently recovered without approachable gross structure damage. Montmorillonite-Bentonite clays therefore come in different varieties depending on which elements are most concentrated in it.

Bentonite clay is chosen for the experiments on edible oil bleaching for its expansible property. The clay when acid activated create more void space through leaching of some components and cation-hydrogen ion exchangeable. In activated state, it acts as catalyst, [3-4]. It has been suggested [4-9] that the greater the concentration of the acid, the greater the catalytic supports. In the chemical industry, there is increase in discoloration and purification when this clay is used. These properties contribute to the use of the clay as component of carbonless copying paper and detergent in paper industries.

Bentonite deposits have been discovered in many Nigerian states, [7-9] cutting across the entire Nigeria. In Yobe, Taraba, Adamawa and Borno an estimate of a probable reserve of over 700 million tones has been indicated. Similarly, over 90 million tones have been reported in Afuze, Ekpoma-Igunebon, Ovibiokhuan and Okpebho, Edo State. Some occurrences have also been reported in Abia, Ebonyi and Anambra States (Table 1). The availability of Bentonite in nearly all the states in Nigeria with the wide range of industrial applications

enhances the attractiveness of the Bentonite processing ventures; hence, improvement in economy of the country.
The present investigation involves the characterization and bleaching property evaluation of Montmorillonite-Bentonite clay collected from Ikorodu-Ibeshi, South-West, Nigeria. The aim of the project was to evaluate the bleaching capacity of Ibeshi Bentonite on soya bean oils (edible oils) before and after acid activation processes while the objective enables the performance evaluation of Nigeria Bentonite for better utilization for production in industries. The results reveal both chemical and physical properties, some of which are the structures characteristics, free fatty acid value, viscosity and elements present in the materials.

Table 1: States and Locations of Bentonite Deposit in Nigeria

S/No	State	Location
1	Edo	Afuze, Ekpoma-Igunebon road, Ovibiokhuan, and Okpebho.
2	Anambra	Awgu
3	Borno	Dikwa, Ngala, New Marte, Mongonu and Mafa
4	Sokoto	Dukamaje and Kalambaina
5	Lagos	Ibeshi LGA
6	Yobe	Fika
7	Taraba	Ibi and Durngel
8	Adamawa	MayoBelwa
9	Abia	Umuahia South Umuahia North
11	Gombe	Pindiga

Table 2: Chemical Properties of Soya bean Oil

Parameters	Amount in Soya bean Oil
Fatty Acid value	0.5 mg KOH/g
Saponification value (S)	16.83 mg/g
Iodine value g /100g	71.32 g/100 g
Refractive index (25°C)	1.645
Viscosity (ŋ) (m^2/s)	39.70 = 3.72 m^2/s 40.91= 3.83 m^2/s

	41.28= 3.86 m^2/s
Peroxide value	52 m mol peroxide/kg sample
pH	6.4 at 23.2°C
pH	6.7 at 24.6°C

Methodology

Sample Collection

The Bentonite clay minerals of Ikorodu were obtained from Ibeshi industrial site, in Lagos state. There was intensive biogenic mixing and irrigation of the bottom place, to the upper few centimeters at the mud. We therefore infer that the influence of macro fauna on the chemistry of the bottom would be limited largely to the surface of the deposit, approximately 10-30 cm in depth. Samples were therefore taken beneath outcrop. Particular attention was paid to mineralogy of the sections of the lumps under reflected light and brushing of the surface. Never-the-less, Bentonite occurs as dark red to tan pink complex growth. Several lumps of samples were taken for subsequent analysis.

Physico-Chemical Analysis of Soya beans

Soya beans used in this study was bought from local market in Iyana, Ota, Ogun state. The oil was extracted in the Chemistry laboratory at Covenant University. All chemicals used were analytical grade. The results of measurements taken for the following were recorded in Table 2. These procedures were carried out for the physico-chemical analyses of both bleach and unbleached oil.

Free Fatty Acid

The oil (10 g, 50 ml) was dissolved in ether and ethanol (25 ml, 95% v/v) in conical flask (250 ml), followed by addition of phenolphthalein indicator (3 drops). This was titrated against KOH (0.1 M) with constantly shaking until a pink color which persists for 15 secs is obtained.

Acid Number / Acid value of oil / Free Fatty Acid = Titre value (X) x Conc. (M) of KOH x 5 /

Mass of oil in gram

Iodine Value of oil / Refractive index/ Viscosity

The extracted oil (0.2 g) was accurately weighed into an iodine flask (25 ml), chloroform (10 ml), iodine solution drained into sample bottles and made up to volume. The refractive index of the soya bean oil was determined using Abbe refractometer. The viscosity of the soya bean oil was determined using a viscometer.

Peroxide / Saponification value

The oil (1 g) was weighed into a clean dry beaker, and then powdered KI (1 g) was added. Glacial ethanoic acid and chloroform (2:1, v/v, 20 ml) were also added. This solution was boiled vigorously for 30 seconds; the content was poured quickly into a conical flask containing KI solution (20 ml, 5%). This solution was titrated with $Na_2S_2O_3$ solution (0.002 M) until yellow color disappeared. Starch (0.5 g) was added, shook vigorously and titrated carefully until blue colour disappeared. Blank solution was measured at the same time for peroxide value determination.

The oil (5 g) was weighed into a conical flask; alcohol (50 ml) was added. Potassium hydroxide was added from the burette by draining for five hours. A blank was prepared by taking KOH (50 ml) drained from the burette. A reflux condenser was connected to the flasks and boiled gently for one hour. The flask and condenser were cooled; indicator (1 ml) was added and titrated against HCl (0.5 M) until the pink color just disappeared, saponification value was then measured.

Pretreatment / Analyses of the raw Bentonite

Bentonite samples were dried at 105° for 4 hours in a drying air oven and then grounded using a mortar and pestle. Other larger samples were crushed using magnetic rotator. A sieve shaker was used to sieve the Bentonite to powder [10]. These samples were ready for chemical analysis, physical-chemical tests, characterization and acid activation.

Bentonite swelling capacity Test / pH

Three (10 g each) portions of dry Bentonite were weighed into a measuring cylinder, followed by addition of distilled water (60 ml). The content in the cylinder was shaken for 10 minutes and left for three days to allow swelling of the clay. The Bentonite samples (3) were left in a beaker; swelling occurred at an increase of 5 ml after 3 days to 15ml. Physico-chemical analyses were carried for both bleach and unbleached oil.

At 6 M

% Swelling capacity, (BC %) = (Vol. of Bleached oil) - (Vol. of Unbleached oil) x 100% / (Vol. of Bleached oil)

(2.276) – (1.504) / (2.276) = 0.34%

7 M

(Bleached oil) - (Unbleached oil) x 100% / (Bleached oil)

(1.63) – (1.504) / (1.63) = 0.08%

8 M

(Bleached oil) - (Unbleached oil) x 100% / (Bleached oil)

(1.649) – (1.504) / (1.649) = 09%

The pH of the Bentonite was determined by dissolving 30 g of the clay into 180 ml distilled water and stirring for few minutes. Electrode, 9024 model microcomputer pH meter (Hanna products), was used.

Both the reference and indicator electrodes were cleaned with tissue paper, then dipped into distilled water (100 ml) for 30 mins, until the reading is pH 7. The indicator electrode was removed from the distilled water and dipped into solution of the sample and stirring continued for few minutes; then the reading was recorded.

Preparation and analyses of acid activation of Bentonite

Acid-activation Bentonite were prepared (from the above raw Bentonite) with sulfuric acid, 6 M, 7 M, and 8 M magnetic stirring for 1 hour at 60° on hot plate. The ratio of Bentonite to sulfuric acid solution was 1 : 2 (w/v). The filtered clay was then dried at 60° to reduce moisture content until the weight was constant. The dried acid-activated Bentonite was kept in sample nylons for study of oil bleaching.

Bleaching of the soya bean oil

The three acid activated Bentonite samples were weighed (10 g) into various beakers labeled 6 M, 7 M, and 8 M respectively. The soya bean oil (20 ml) was added into the same container as the Bentonite. The solution was stirred for 1 hour using hot plate, after which it was filtered with double-layer filter papers. The bleached soya bean oils were ready for further tests.

Evaluation of bleaching performance / Bentonite GC/MS Analysis

The maxima absorbance wavelength and absorbance values of chlorophyll and carotene were obtained by using a UV-VIS spectrophotometer. Bleaching capability, BC was evaluated by monitoring the absorbance at every maxima wavelength, applying the following formula.

$$BC = A^{o}_{\lambda}\,(\text{Neutralized oil}) - A_{\lambda}\,(\text{Bleached oil}) \times 100\% \,/\, A^{o}_{\lambda}\,(\text{Neutralized oil})$$

Here, A_{λ} is the optical density at wavelength λ.

The procedure employed in this work, for chemical analysis of Bentonite are Gas Chromatography Mass Spectrometry (GC/MS) and Atomic Absorption Spectroscopic (AAS) analytical techniques. The results obtained for base-metal containing clay minerals of Ibeshi-Ikorodu South Western Nigeria are as shown in Figures 2-9 and Tables 3-4.

Results and Discussion

The compositional analysis from atomic absorption spectroscopy characterization, showed the ratio of Na_2O:CaO be 0.24-0.30, a value less than one, indicative the presence of montmorillonite. Furthermore, the SiO_2: Al_2O_3 between ratio of 1.12-1.50 (greater than one) is suggestive a clay suitable for bleaching. However, associated clay minerals, for example Zeolite can be present at Ibeshi because of their similar chemical composition [3].

Laboratory tests for the evaluation of acid activation and bleaching performance of Ibeshi clay gave percent colour reduction of 9% increased to 28% after 8 M H_2SO_4 activation, a value moderate for effective bleaching. The ion is thought of as being derived from orthosulicic acid H_4SiO_4 during acid activation. This is possible because of the octahedral layer (Figure 2). The Na^+, the exchange takes place in aqueous solution, so that H^+ is available as a competitor at exchange sites. The exchange capacity is observed at pH 7 or higher, but H^+ interference may occur in some systems. The clay has bleaching action.

In Montmorillonite, there is substitution in the octahedral and tetrahedral units giving, excess negative charges, Figure 1, unsatisfied valences on edge of units. It has been shown [11-12] that a 100 g sample of Montmorillonite when treated with a NaCl solution so that all exchange site are occupied by sodium ions, and then placed in a series of strong KCl solution, sodium ions appear in moderate concentration in the firsts treating solution, and lesser concentration in succeeding KCl solutions.

Physico-Chemical Parameters of Oil

The physical state of soya bean oil is liquid in nature; the colour is dark brown and odorless. Other physical properties of soya bean oil are listed in Table 2.

Analyses of bleaching capacity

According to the absorbance read on UV-VIS analysis, Figures 2-6, each of the four samples, the absorbance of the unbleached was the lowest; this is because of the compounds present during acid activation of Bentonite. At 6 M the absorbance recorded was very high compared to the peaks at 7 M and 8 M, which further explains that 6 M is the most suitable molarities of acid activation for bleaching to occur. The optimum acid concentration for industrial bleaching could be 6 M H_2SO_4.

GC/MS

In the GC-MS analysis, Figures 7-9, of the raw Bentonite, the spectra show the dominant components present in the raw Bentonite: SiO_2, Al_2O_3, Fe_2O_3, MgO, CaO, Na_2O, K_2O, TiO_2, MnO_2 and P_2O_5. Results in Tables 3-4, revealed the presence of other organic compounds: 8% vitamin E (α-Tocopherol), which is used in body creams to remove scars [3], stigmasterol was 9%, and this is similar to animal cholesterol use to cure various types of cancer such as: colon, ovarian, breast and prostate cancers. This is to say that Bentonite can also be described as a "Healing balm", asides its bleaching property for vegetable oils. However, bleaching of the oils by adsorption could have removed some pigments. The GC-MS spectra of the oil showed a change in the total percentage of the components in the unbleached and bleached oil. In the unbleached the total percentage of the components present is higher because of the other compounds present while the total percentage in the bleached is reduced.

Conclusion

The exchange capacity at pH 7 makes the system none acidic. Absorbance capacity of other compounds by Bentonite from the unbleached soya bean oil is prominent in the work. The bleaching performance evaluation confirmed that the Bentonite clay minerals of Ibeshi-Ikorodu South-Western Nigeria have moderately bleaching action. The availability of Bentonite in Nigeria and the wide range of industrial applications; enhance the attractiveness of the Bentonite processing ventures. This could add values to export commodities of the country. The scope of studies of Montmorillonite/Bentonite ore-bodied areas in Nigeria should be expanded to an extent where there is a need for individuals with competence in separate areas: geochemistry, biotechnology, mineral processing, material engineering, and medicinal sciences.

Acknowledgement

We wish to acknowledge Owoeye, Taiwo Felicia for her assistance during laboratory work.

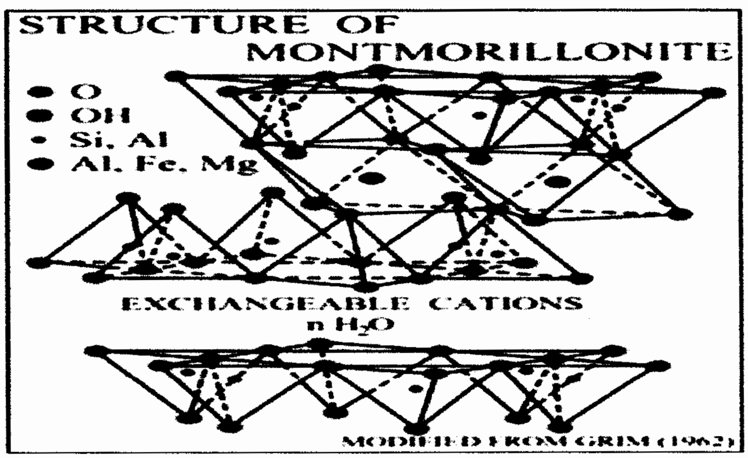

Figure 1: Chemical structure of Montmorillonite

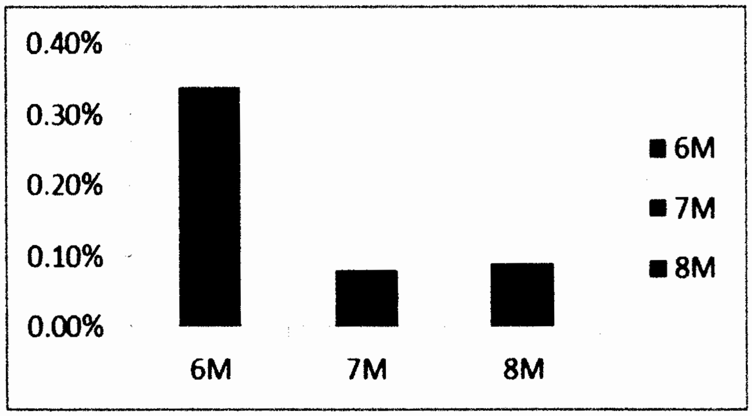

Figure 2: Bleaching efficiency of the acid-activated Bentonite

Figure 3: UV-VIS analysis of unbleached oil

Figure 4: UV-VIS analysis of bleached oil at 6M

Figure 5: UV-VIS analysis of bleached oil at 7M

Figure 6: UV-VIS analysis of bleached oil at 8M

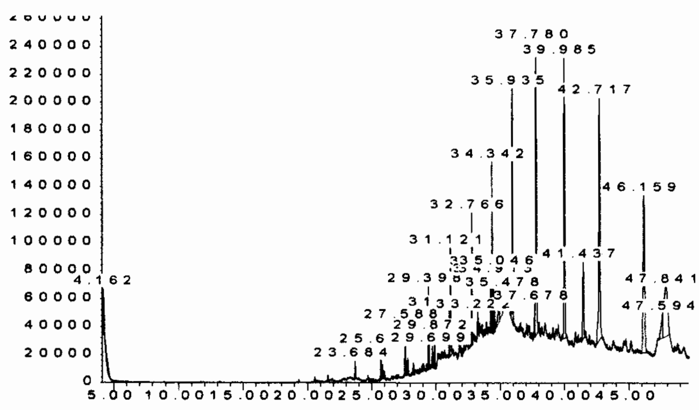

Figure 7: Image of GC-MS analysis on raw Bentonite

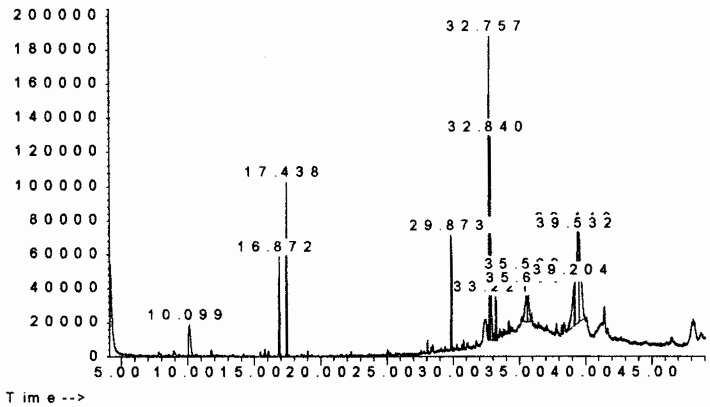

Figure 8: Image of GC-MS analysis on unbleached oil

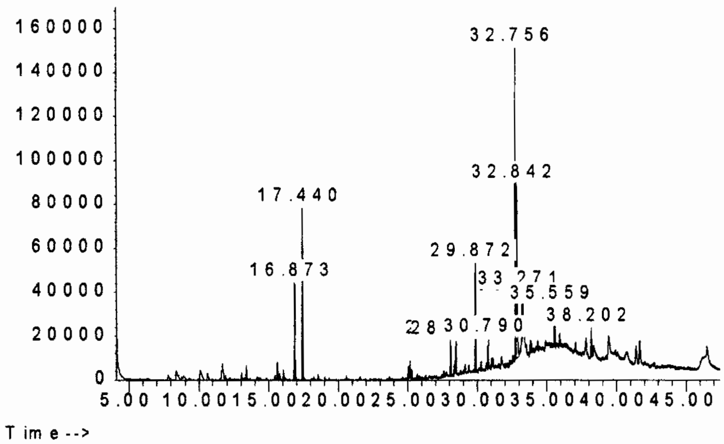

Figure 9: Image of GC-MS analysis on bleached oil

Table 3: Ikorodu/Ibeshi Bentonite-Montmorillonite Data Path

Data Path : C:\msdchem\1\methods\THC.M\
Data File : Folake 1 mm.D
Acq On : 25 Mar 2014 15:26
Operator: MEJIDA/ACHEM
Sample : Folake 1
Misc :
ALS Vial: 3 Sample Multiplier: 1
Search Libraries: C:\Database\NIST08.L Minimum Quality: 90
C:\Database\NIST11.L Minimum Quality: 90
Unknown Spectrum: Apex
Integration Events: ChemStation Integrator - events.e

Pk#	RT Area%	Library/ID	Ref#	CAS#	Qual
1	16.875	8.31 C:\Database\NIST08.L			
		2, 4-Decadienal, (E,E)-	24684	025152-84-5	91
		2, 4-Decadienal, (E, E) -	24685	025152-84-5	72
		2, 4-Nonadienal	17289	006750-03-4	64
2	17.442 14.78 C:\Database\NIST11.L				
		2, 4-Decadienal, (E,E)-	25130	025152-84-5	87
		2, 4-Decadienal, (E, E) -	25129	025152-84-5	87
		2, 4-Nonadienal	17515	006750-03-4	86
3	28.079 2.68 C:\Database\NIST11.L				
		1, 1, 1, 5, 7, 7, 7-Heptamethyl-3,3-bis trimethylsiloxy)tetrasiloxane	(225661	038147-00-1	72
		2-(2', 4', 4', 6', 6', 8', 8'-Heptamethy Ltetrasiloxan-2'-yloxy)-2, 4,4,6,6, 8, 8, 10, 10-nonamethylcyclopentasiloxane	242215	145344-72-5	28
		3-Isopropoxy-1, 1, 1, 7, 7, 7-hexamethy l-3,5,5-tris(trimethylsiloxy)tetrasiloxane	240263	071579-69-6	25
4	28.462 2.65 C:\Database\NIST11.L				
		2-Isopropyl-5-oxohexanal	28978	015303-46-5	40
		Oxirane, dodecyl-	71312	003234-28-4	16
		2-Piperidinone, N-[4-bromo-n-butyl]	88460	195194-80-0	12
5	29.870 9.71 C:\Database\NIST08.L				
		Hexadecanoic acid, methyl ester	113690	000112-39-0	97
		Pentadecanoic acid, 14-methyl-, me thyl ester	113705	005129-60-2	97
		Hexadecanoic acid, methyl ester	113682	000112-39-0	97
6	30.791 1.91 C:\Database\NIST11.L				
		3-Isopropoxy-1, 1, 1, 7, 7, 7-hexamethy l-3,5,5-tris(trimethylsiloxy)tetra siloxane	240263	071579-69-6	59
		Hexasiloxane, tetradecamethyl-	228692	000107-52-8	50
		Octasiloxane, 1,1,3,3,5,5,7,7,9,9, 11, 11, 13, 13, 15, 15-hexadecamethyl-	240341	019095-24-0	40
7	32.754 27.54 C:\Database\NIST08.L				
		9, 12-Octadecadienoic acid, methyl ester	132259	002462-85-3	99
		9, 12-Octadecadienoic acid, methyl ester, (E,E)-	132278	002566-97-4	99
		9,12-Octadecadienoic acid (Z,Z)-, Methyl ester	132273	000112-63-0	99
8	32.840 19.42 C:\Database\NIST08.L				
		6-Octadecenoic acid, methyl ester, (Z)-	133721	002777-58-4	99

	9-Octadecenoic acid (Z)-, methyl e ster	133717	000112-62-9	99
	Cis-13-Octadecenoic acid, methyl e ster	133713	1000333-58-3	99
9	33.223 3.08 C:\Database\NIST11.L			
	Methyl stearate	143127	000112-61-8	87
	Methyl stearate	143126	000112-61-8	64
	Methyl stearate	143128	000112-61-8	50
10	33.269 3.39 C:\Database\NIST11.L			
	Squalene	215930	000111-02-4	38
	2, 6, 10-Dodecatrien-1-ol, 3, 7, 11-TR imethyl-, (Z,E)-	79440	003790-71-4	38
	2, 6, 10-Dodecatrien-1-ol, 3, 7, 11-TR imethyl-	79429	004602-84-0	38
11	35.558 3.37 C:\Database\NIST11.L			
	1, 1, 1, 5, 7, 7, 7-Heptamethyl-3, 3-bis (trimethylsiloxy)tetrasiloxane	225661	038147-00-1	50
	Heptasiloxane, 1,1,3,3,5,5,7,7,9,9 , 11, 11, 13, 13-tetradecamethyl-	235668	019095-23-9	38
	3-Isopropoxy-1, 1, 1, 7, 7, 7-hexamethy L-3, 5, 5-tris(trimethylsiloxy)tetra siloxane	240263	071579-69-6	37
12	38.201 3.16 C:\Database\NIST11.L			
	Cyclononasiloxane, octadecamethyl-	242431	000556-71-8	72
	1, 1, 1, 5, 7, 7, 7-Heptamethyl-3, 3-bis (trimethylsiloxy)tetrasiloxane	225661	038147-00-1	50
	Hexasiloxane, tetradecamethyl-	228692	000107-52-8	47

Table 4: THC.M. Data Path

Wed Mar 26 10:47:56 2014
Area Percent Report
Data Path : C:\msdchem\1\methods\THC.M\
Data File : Folake 1 mm.D
Acq On : 25 Mar 2014 15:26
Operator: MEJIDA/ACHEM
Sample : Folake 1
Misc :
ALS Vial: 3 Sample Multiplier: 1
Integration Parameters: events.e
Integrator: ChemStation
Method : C:\msdchem\1\methods\THC.M
Title :
Signal : TIC: Folake 1 mm.D\data.ms

Peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	16.873	2224	2235	2256	BB 2	43155	1329182	0.19%	8.315%
2	17.440	2316	2334	2345	BV	77464	2362689	53.66%	14.780%
3	28.079	4173	4193	4204	BV 2	16190	427760	9.72%	2.676%
4	28.463	4250	4260	4280	BB 3	14706	423150	9.61%	2.647%
5	29.872	4485	4506	4524	BV	49105	1552677	35.27%	9.713%
6	30.790	4656	4667	4675	PV 2	13066	305601	6.94%	1.912%
7	32.756	4990	5010	5018	BV 2	140557	4402859	100.00%	27.543%
8	32.842	5018	5025	5057	VV 5	78596	3105088	70.52%	19.424%
9	33.223	5057	5092	5096	PV 5	18900	491706	11.17%	3.076%
10	33.271	5096	5100	5110	VV 4	19439	541519	12.30%	3.388%
11	35.559	5482 5	500	5513	BV 2	18318	537937	12.22%	3.365%
12	38.202	5941	5962	5981	BB 3	13077	505469	11.48%	3.162%
		Sum of corrected areas:			15985636				

THC.M Wed Mar 26 10:48:33 2014

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