



The Chemical, Mineralogical, Structural and Surface Properties of Selected Ugandan Clays and their Application in Bleaching Oil

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INTRODUCTION

1.1 Background

For a long time, clays have been used to clarify mineral and vegetable oils (Nutting, 1933; Siddiqui, 1968). Whereas vegetable oils for human consumption are clarified using bleaching earths (Adekeye and Bale, 2008; Ajemba et al, 2012; Kamal et al, 2011; Mustapha et al, 2013; Patterson, 1992; Proctor, 1988; Salawudeen et al 2007; 2014; Taylor, 1984; Zchau, 1981), other oils can be clarified using peroxides.

Clays have been used for healing, cosmetic and nutritive purposes since ancient times. Healing clays help the body rid of toxins. Bentonites gained popularity for internal and external detoxification (Aufreiter et al, 1997). Clays as antibacterial agents were evaluated (Hui et al, 2005; Tong et al, 2005; Williams and Hydal, 2010) and those used in medicines are bentonites (Carretero and Lagaly, 2007) kaolinites are used for digestive upsets absorbing acid and gases.

Geophagic clays have high concentrations minerals as silica, calcium, magnesium, iron, sodium, potassium (Ekosse and Anyangwe, 2012; Gichimbi et al, 2012) so they act as natural sources of minerals. Clay minerals are

traditionally mixed with water to form pastes or gels applied externally for cosmetic or skin protection (Gomes et al, 2007, Carretaro et al, 2002, 2007) because of their high adsorptive and absorptive capacities. Clays were used to clarify mead until recently when a brand sold in UK was found to introduce arsenic and lead in the alcohol. Clays and clay minerals remove organic pollutants from water (Tahir et al, 2013; Zadaka et al, 2007) and aflatoxins (Jaynes et al, 2007).

Vegetable oils contain many impurities including peroxides, pesticide residues and heavy metal ions which can be harmful to man as well as carotenes, tocopherols, phytosterols and squalenes which are nutritive (Cho-Ah-Ying, 1991; Goh, 1986; Peymaneh et al, 2014; Salawudeen et al, 2007; Usman et al 2012, 2013); tocopherols and tocotrienols which have vitamin E activity in the human body (Wang, 1999); phytosterols which have pharmaceutical effects (Khor and Chieng, 1997). Removal of carotenes, gums, pigments and peroxides during the bleaching process renders the oils less harmful and nutritive.

In Uganda clays used in the vegetable oil industries are imported from South Africa, China, USA, or UK; spending foreign currency on

materials that can be prepared from local earths in Uganda. As all oil industries are private and there is lack of legislation enforcing use local materials, the owners of the industries cannot venture in research because it is expensive and will lower the savings.

The local populace in many parts of this country produce oils by pressing simsim, groundnut, date palms, cashew nut or/and sunflower paste, and use it without bleaching. This poses a threat of diseases arising from consumption of heavy metals, peroxides and pesticide residues yet local clays can be used to remove impurities from oils (Bakhtyar et al, 2011; Bastos-Andrade et al, 2014; Mbah, et al, 2005; Naeem and Ahmad, 2008; Mustapha et al, 2013; Nir et al, 2006; Nwabanne and Ekwu, 2013; Salawudeen et al, 2007; 2014; Topkafa et al, 2013; Ujeneza et al, 2014; Usama et al, 2012; 2013; Valenzuela-Diaz and Souza-Santos, 2001; Veniale et al, 2007). Clays from Kajansi were successfully used to bleach cottonseed oils (Dandy, 1967), however, clays from Kajansi, Kitetikka, Koki and Mutaka have been developed to make bricks and tiles (Kirabira et al, 2005; 2004; Nyakairu and Kahwa, 1998), not bleaching earths.

The economy of Uganda is largely agricultural, needs to be diversified through value addition. The growing of oil seeds (such as cotton, groundnuts, sunflower, cashew nuts, simsim, pumpkins and palm oil dates started many years ago to boost the oil production in Uganda. Palm oil growing estates have been set up on the Kalangala islands. Several oil processing factories have cropped near towns up such as Mukwano, Gulu nanak, BIDCO, Nile agro processing and Mawokota soap works and the sales of oil made in Uganda is quite high. In all these oil industries, bleaching earths are imported, though they could use what is local since all other in-puts are local and reduce on expenditure.

Importation entails spending the scarce foreign exchange, in addition to raising production costs while promoting foreign industries from which the earths are imported. And this denies Ugandans employment opportunities in such industries

which would locally produce the earths since they will not prevail.

As the bleaching earths are imported, there must be tendencies of lowering the standard of oil on the market in order to offset production costs. This may lead to consumption of low grade oils containing heavy metals, peroxides and pesticide residues by unsuspecting Ugandans.

1.2 Statement of the problem

Uganda is endowed with a big diversity of natural resources which would be very instrumental in growth and development of the vegetable oil industry. The country is rich in clay deposits as well as plants which bear fruits that produce vegetable oils on commercial scale. However, these resources are largely untapped, neglected or utilized haphazardly because the science of using local clays to bleach is limited.

In addition, scientific data needed to develop bleaching earth mines is limited despite the fact that the volcanic clays that are used to produce bleaching earths are available in Uganda in areas of past volcanic activity. Further more, little is known about the quality of Ugandan clays in bleaching the oils. Similarly, the quality of oils bleached using Ugandan clays is unknown. The possibility of developing local clays to bleaching earth can result from well documented research. It is the aim of this study to furnish such data that is crucial to developing Ugandan bleaching earths. In this respect therefore, both clays and vegetable oils will be analyzed fully before and after interacting with each other to characterize a Ugandan bentonite.

1.3 Significance of the study

The results from this research will provide alternative ways of utilizing Uganda's local resources by availing data on the nature and means of converting clays of Uganda to industrial bleaching earths and quality of oils bleached using local earths, which can be used in the local industries here and/or be exported.

The data will be used for setting up bleaching earth industries thereby increasing on the job

market and tax base for the government. The information will save Ugandans from using raw or partially bleached oils thereby presenting a possibility of reducing health hazards caused by consuming heavy metals, peroxides and pesticide residues in oils.

1.4 Justification of the study

The characteristics of Ugandan bleaching clays remain unknown, yet these clays are on worldwide demand. So there is urgent need for comprehensive study to elucidate the surface, structural, mineralogical, chemical and bleaching properties of natural clays from Uganda.

Investors may get interested in developing the bleaching earth industry when knowledge from this research is disseminated thereby increasing the job market.

1.5 General objective

The general objective of the study is to determine the bleaching, chemical, mineralogical, structural and surface properties of selected clays from Central and Eastern Uganda, and the quality of vegetable oils bleached with them. The general objective will be achieved from the following specific objectives.

1.5.1 Specific objectives

- i. To determine elemental, mineralogical and structural properties of selected clays from Eastern and Central Uganda.
- ii. To bleach vegetable oils using calcined and activated selected and spent bleaching clays at differing temperatures; record absorbance, turbidity and refractive indices of bleached oils.
- iii. To determine surface acidity of clays by equilibrating clay fractions with 1-aminobutane and determining free 1-aminobutane left unbound.
- iv. To determine quality of the bleached vegetable oils by determining iodine, free fatty acid, acid, trace metals and peroxide values of oils.

- v. To isolate and characterize the clay with highest bleaching capacity using IR, DTA and XRD patterns.

LITERATURE REVIEW

2.1 Clays

Clays belong to a group of minerals collectively called alumino-silicates. They contain aluminium oxide and silicon dioxide as universal minerals (Bailey, 1980; Hamza, 1966; Nguetrikam et al, 2008; Woumfo et al, 2007) and have special properties ranging from catalytic, sorption, ion exchange, discoloring, filter aid to catalyst support (Murray, 2000; Papp, 1971).

Methods of preparing bleaching earths were described by many authors (Dandy, 1965; Motlagh et al, 2011; Nguetrikam et al, 2008; Nutting, 1935; Welsberger, 1968; Woumfo et al, 2007). Clays of Uganda are used for bricks, tiles and/or ceramics (Kirabira et al, 2005;2004; Mathers and Mitchell, 1992; Nyakairu and Kahwa, 1998); they could be used to discolor crude oils or as filter aids if researched on.

Clays were classified, (Longstaffe, 1981) and used to bleach (Adekeye and Bale, 2007; Boki and Ohno, 1989; Falaras et al, 2000; Kaufmann and Mukherjee, 1967; Liew, 1994; Morgan, 1985; Naeem and Ahmad, 2008; Nir et al, 2006; Norris, 1982; Patterson, 1992; Peymaneh et al, 2014; Salawudeen et al, 2007; 2014; Taylor, 1984; 1989; Usama et al, 2012; Valenzuela-Diaz and Souza-Santos, 2001). Soy bean, sunflower-seed, cottonseed, Olive, corn, soybeans and all other seed oils fall into the general family of oils known as vegetable oils. Vegetable oil is one of the liquid substances that are extracted from the vegetables. Vegetable oil is almost entirely made up of the fat of the vegetable. It can be liquid at room temperature, as in the case of corn, cottonseed, soybean, or olive oil.

Four basic steps are used to refine oil: neutralization and separation, bleaching and deodorizing (Siddiqui, 1968; Zschau, 2001). Diatomaceous earth, clays, peroxides or carbon is added to bleach and adsorb the dark colored impurities in the oil in order to give it a clear color

(Adekeye and Bale, 2007; Al-Zaharani et al, 2000; Boki and Ohno, 1989; Falaras et al, 2000; Kaufmann and Mukherjee, 1967; Liew, 1994; Morgan, 1985; Naeem and Ahmad, 2008; Norris, 1982; Patterson, 1992; Salawudeen et al, 2007; Taylor, 1984; 1989; Usama et al 2012;2013; Valenzuela-Diaz and Santos-Souza, 2001). Some of oils may have a color that is objectionable to a consumer. Thus, the oil needs to be bleached to improve its color quality. To this end, a great many oils are commonly treated with bleaching clays to reduce oil color values by adsorptive purification (Adekeye and Bale, 2008; Ajemba et al, 2012; Didi et al, 2009; Rich, 1964; Liew, 1994; Mokaya, 1983; Swoboda, 1985; Topkafa et al, 2013; Ujeneza et al, 2014; Valenzuela-Diaz and Santos-Souza, 2001).

Clays were classified into phyllosilicates and layers silicates. (Bailey, 1979) and have for long been used as clarifying agents for vegetable oils for human consumption (Nutting, 1935; Srasra, 1989; Wiederman, 1981), filling white paper, drilling fluids, geophagy (Ferrell, 2008) as well as making bricks, tiles, sanitary wares, cups, plates, insulation porcelain Kirabira et al, 2005 and curative (Maigann and Pareilleux, 1971; Ghinci et al, 2009). Curative clays are bentonites for Buruli ulcers, skin bacterial and fungal infections (Williams and Hydral, 2010, Wlison, 2003) and kaolinites for acid flux, constipation, bloating, gas (Ferrell, 2008).

It is this background of immense diversity of applications that drives the need to study the surface, structural, compositional and bleaching characteristics of natural clays in Uganda.

2.2 Structural studies of clays

Clays have different but inter-related structures. These materials are alumino-silicates of wide range of compositions and utility. Clays never attain the cage structures although their crystalline structures can be very large (Lagaly, 1995; McGraw, 1993; Souza-Santos, 1989).

Clays are sub-divided in kaolinites and montmorillonites or bentonites basing on whether they are formed by alteration acid-ingenious rocks

or alkali-intrusive magmatic rocks or ashes respectively (Christidis, et al 1993). Kaolinites are different from smectites but show resemblance to clays related to them by diagenesis (Bailey and Brindley, 1979; Gates, 2002). Clays are known to be natural, earthy, fine-grained materials which develop plasticity when mixed with a limited quantity of water (McGraw, 1998). They are composed principally of silica, alumina and water often with iron, alkali or alkaline earth metals (Souza-Santos, 1989).

2.3 Mineralogy of clays

Clay minerals which are widely studied and used are broadly categorized in two groups, kaolinites and smectites. The kaolinites include kaolinite, halloysite, and dickite (Longstaffe, 1981); and the smectites include montmorillonite, nontronite, saponite and hectorite (Bailey and Brindley, 1979). Whereas the kaolinite minerals consist of a single tetrahedral silica sheet and single octahedra alumina sheet forming repetitive sheets in infinite dimensions, smectites may consist of a single silica tetrahedral between two or more alumina octahedral sheets (Altaner, 1990; Bailey, 1980; Christidis et al, 1998). In Uganda, clay deposits at Mutaka, Kajansi and Kitetikka were characterized to be kaolinite largely (Reedman, 1969, Nyakairu et al, 2001; Kirabira et al, 2005)

2.4 Structural properties of clays

Kaolinites differ from smectites because they form under different diagenetic conditions. The differences in physical and chemical properties are related to their origin. Kaolinites form from igneous acid diorites which are structurally different from alkaline intrusive magmatic turfs from which smectites form (Mathers, 1998). Smectites consist of two silica tetrahedral sheets for every one octahedral sheet yet kaolinites consist of one tone sheets of tetrahedral to octahedral sheets (Souza-Santos, 1989; Tyagi et al, 2006). DTA, IR and XRD have been used to distinguish between kaolinites and smectites. Differential thermal analysis was used for the determination

of the kaolinite disorder degree according to decomposition peak temperature obtained from DTA curves (Naeem and Ahmad, 2008; Vaculikova et al, 2011).

2.5 Applications of clays

Owing to their structural properties, clays are used in many ways including construction, foundry, insulation (Kirabira et al, 2005) adsorption, (Christidis and Dunham, 2003; Weikamp, 1991) filter aids (Arioua and Daud, 2011; Folleto et al, 2006; Karger, 1992; Nguetrikam et al, 2008), catalysis (van Bekkum, 1991).

2.5.1 Adsorptive uses of clays

Whereas kaolinites are rarely used to bleach, acid-leached smectites are widely used to clarify oils. The bleaching of soybean oil was done using adsorptive bentonite clays (Joy et al, 2007; Wiederman, 1981). Interactions among oil components during adsorption were studied and decrease in carotenoids and Peroxides values observed (Adekeye and Bale 2008; Aroua and Daud 2011; Boki, and Ohno, 1989; Cho-Ah-Ying, 1991; Dandy, 1965; Foletto et al, 2011; Liu et al, 2008; Liew, 1994; Morgan, 1985; Naeem and Ahmad, 2008; Proctor, 1988; Sabah and Majdan, 2009; Salawudeen et al, 2007; 2014; Sarier, 1988; Shahidi, 1997; Taylor, 1989; Toro-Vazquez., 1991; Usama et al, 2012; 2013). The surface area of a sepiolite was determined using carbon dioxide adsorption isotherms (Dandy, 1967).

2.6 Geology of clays

The transformation of bentonites to illites and kaolinites under the hydrothermal alteration decreases the smectite content via replacement by kaolinite or halloysite and precipitation of various silica polymorphs, carbonates, sulphates and sulphides (Christidis et al, 1995; 1993).

The factors which favor transformation of bentonite to illite are temperature, (Harvey Blatt, 1971; Harvey, 1991; Hower, 1976); pressure (Velde, 1986); potassium availability, (Howard,

1985); pore fluid chemistry (Roberson, 1981); and smectite composition (Eberl, 1978). The transformation of bentonite to illite involves the release of chemical elements which participate in formation of quartz, chlorite and/or kaolinite by products (Hower, 1976). Most Ugandan clays have been transported, found in river valleys consisting of kaolinites and smectites (Mathers and Mitchell, 1998; Nyakairu et al, 2001; Reedman, 1971).

Many bentonite deposits are formed by in-situ alteration of airborne and water transported volcanic ash in shallow lake and sea water (Altaner, 1990; Jeans 2000; Elzea, 1990). Primary bentonites form by in situ hydrothermal alteration of ultrabasic volcanic glass, ash or feldspar (Mathers and Mitchell, 1992). Both smectite and kaolinite rich clays can result from tropical weathering and the acid or basic nature of the environment is the determining factor for the clay type formed (Brigatti et al, 2006; Charnley, 1989; Guggenheim et al, 2006; Komadel and Madejova, 2006; Lagaly et al, 2006; Madejova, 2003; Murray 1991).

X-ray diffraction, infrared, thermal differential analyses have been used to identify different clay minerals (Bailey, 1980; Christidis et al, 1997; 1993; Farmer 1979; Knechtel, 1962; Komadel, 2001; Russell, 1979; Wilson, 1974). Characterization of Kashmir bentonite was carried out using XRD, DTA and CEC (Naeem and Ahmad, 2008).

2.7 Surface properties of clays

Clays exhibit unique surface properties like acidity, high surface areas, cation exchange, bleaching capacity and adsorption (Balaras, 2000; Barrer, 1989; Christidis, et al 1995; Gomiz, 1992; Hassan and El-Shall, 2004; Pinnavaia, 1983; Srasra, 1989) . Each of these properties is going to be treated separately for convenience.

2.7.1 Acidity of clays

It was reported that the aluminium ion to be four coordinate must have replaced a silicon (IV) ion in the clay matrix (Weiss, 1948). When leached,

the aluminium (IV) ions are easily removed leaving a structure containing hydrogen and hydroxide ions substituting for the aluminium (IV) ions (Weil-Malherbe and Weiss, 1948). Surface hydrogen and hydroxide ions, play a big role in bleaching (Hofmann, 1935; Nutting, 1931; Srasra and Trabelsi-Ayedi, 2000). Presence of water (Hansford, 1947); and acid properties (Alemdragloru et al, 2003; Greenall, 1948; Noyan et al, 2007; Mills et al, 1950; Zhansheng et al, 2006) are strongly related to bleaching and cracking capacity of clay matrices.

Certain organic ammonium complexes of bentonites in different organic liquids show striking properties (Barrer, 1989; Jordan, 1949; Siddiqui, 1968; Tanabe, 1970). It has been explained that bentonites bleach oils by reacting with impurities forming organophilic complexes. Montmorillonite and kaolinite are ubiquitous in nature and can be intercalated using $[\text{Nb}_6 \text{Cl}_{12}]^{2+}$ or $\text{Al}_{13}\text{O}_4(\text{OH})_{24}(\text{H}_2\text{O})_{12}$ to give expanded layers with improved diffusion, sorption and catalytic properties (Cotton, 1988).

2.8 The effects of bleaching on oils

Degumming and bleaching was observed to remove gums, fatty acids, trace metals, pigments and peroxides (Cho-Ah-Ying, 1991; Christidis, et al, 1997; Folleto et al, 2006; Proctor, 1988; Taylor, 1984). Oxidation products in the crude vegetable oils (Zchau, 1981; Zschau, 1983; 2001) are removed by adsorption on the active surface of the bleaching earth to improve color and stability of the final oil and improved its quality (Nguetrikam et al, 2008; Shahidi, 1997; Zhang, 1992). The industrial bleaching of vegetable oils using clays was reviewed (Norris, 1964; Kaufmann and Mukherjee, 1967).

Degumming and bleaching, however, remove minor constituents like carotenes, tocopherols, phytosterols and squalenes which have nutritional aspects (Cho-Ah-Ying, 1991; Goh, 1991; Liu et al, 2008). Similarly, tocopherols and tocotrienols have vitamin E activity in the human body (Wang, 1999). Major phytosterols in palm oil include campesterol, stigmasterol and β -sitosterol, have

pharmaceutical effects but are removed by bleaching.

Acid-activated clay adsorbed carotenes (Meesuk and Vorasith, 2006; Sarier, 1988; Wei et al, 2004) and phosphorous ions (Kheok and Lim, 1982; Sabah and Celik, 2005). Adsorption to clay active sites takes place by formation of hydrogen bonding with the Broensted sites or coordination bonds with Lewis acid sites forming stable carbonium ions.

2.9 Surface acidity of clays modified by heating and acid-leaching

Whereas koalinites are used for tile, brick, and pottery making (Mathers, 1992), smectites are used as selective adsorbents (Barrer, 1989), bleaching earths (Christidis et al 1997; Dandy, 1967; Siddiqui, 1968; Wiederman, 1981), catalysts (Pinnavaia, 1983b) and molecular sieve making (Barrer, 1978); pillared clays (Lahav, 1978; Plee and. Gatineau, 1987) organoclays (Mills et al, 1950) foundry and ceramic works (Bradley, 1951; Brindley, 1978).

Clays change their surface properties when leached with acids or heated (Noyan et al 2006). Commercially used acid-leached clays are commonly smectites. Acid leached smectites have been used in oil industries as catalysts, filter aid and bleaching agents (Fahn, 1976; Falaras et al, 2000; 1999; Kooli, 1997; Shahidi, 1997; Siddiqui, 1968; Srasra, 1989; Toro-Vazquez., 1991; Zhang, 1992).

Acid-leached clays show different characteristics from their raw counter parts; for example they show higher acidity and bleaching capabilities than raw clays. For clay to bleach, it must interact with the medium in which the materials or impurities to be removed are present. The interactions of clay surfaces with gases or liquids which are used in adsorption and catalysis are governed by the acid-base strengths (Jacobs, 1984; Tanabe, 1970; Tanabe, et al, 1989; Alemadrogrolu et al, 2003). Clay minerals have been shown to possess attractive properties as solid acids (Gonzalez-Parada, 1991; Ravichandran and Sivasankar, 1997; Sarikaya et al, 2003;

Tanabe, 1970); and these properties are associated with acidity brought about by Broensted and Lewis centers.

The adsorption of 1-aminobutane on clays studied produces adsorption isotherms of the first class type according to the Brunauer classification (Gregg; and Sing, 1982). The chemical interaction between the adsorbate and surface is consistent with the Langmuir

adsorption isotherm equation:

$$c/n = (1/Knm) + c/nm \dots\dots\dots 2.1$$

where c is the concentration of substrate in solution that is in equilibrium with the adsorbed substrate, K is the kinetic equilibrium constant of adsorption, n is the amount of adsorbed substrate per gram of solid, and nm is the monolayer coverage, which corresponds to the theoretical amount of solute that covers all the sites in 1.0 g of sample.

2.10 Adsorptive processes exhibited by clays during the bleaching of oils

There are two types of adsorption processes namely; the physical adsorption which is weak with van der Waals’s forces (Laidler, 1987) and chemisorption process which was first considered by Langmuir, (1916) and showed that the adsorbed molecules are held to the surface by covalent forces. He considered the distribution of adsorbed molecules over the surface in form of unimolecular layer. For the dynamic equilibrium between adsorbed and free molecules he proposed the relation;

$$\frac{P}{x/m} = (1/a)(b/a)^p \dots\dots\dots 2.2$$

Where p is equilibrium pressure for substance adsorbed; x is amount of substance adsorbed; and m is amount of adsorbent; a and b are constants.

From equation 2.2, Freundlich (1926) developed an equation relating adsorption to residual solute concentration shown below:

$$x/m = KC^n \dots\dots\dots 2.3$$

Where C is the amount of residual substance; K and n are constants.

As absorbance measurements are taken in all experiments involving the bleaching processes,

the relative quantity of pigment adsorbed, x and the residual relative quantity at equilibrium, Xe are obtained from equations 2.2 and 2.3 (Boki, 1994; Wu and Li, 2009).

$$x = (Ao -At)/Ao . \dots\dots\dots 2.4$$

$$xe = At/Ao = 1- x \dots\dots\dots 2.5$$

where Ao is absorbance of unbleached crude oil and At is absorbance of bleached oil at time t.

Using equations 2.4 and 2.5 and writing Xe instead of equilibrium pressure, P and the residual concentration, C; equations 2.2 and 2.3 have been rearranged as follows (Achife and Ibemesi 1989 Liu et al 2008; Ujeneza et al, 2014);

$$xe_{/(x/m)} = 1/a + (b/a) xe \dots\dots\dots 2.6$$

$$x/m = Kxe^n \dots\dots\dots 2.7$$

The Langmuir and Freundlich equations to the adsorption isotherms were applied to adsorption of β - carotenes on montmorillonite, activated clay and sepiolite from alkali- refined rapeseed and soybean oils (Ajemba et al, 2012; Boki et al, 1994, Ujeneza et al, 2014). The Langmuir and Freundlich equations to adsorption isotherms were further applied to the bleaching of crude sunflower seed oil with bentonite and showed that absorbance of bleached sunflower oil decreased as the concentrations of bentonite increased with increase in temperature between 60 and 90°C (Lin and Yoo, 2007; Liu et al 2008; Mustapha et al, 2013; Topallar, 1998; Ujeneza et al, 2014).

2.11 Recycling of bleaching earths

Spent bleaching earths are by-products of vegetable oil refining industries. The refining process of salad oils uses many tones of bleaching earths and spent bleaching earths are commonly disposed at landfills. Landfills disposal of spent bleaching clays is an environment hazard; oils catch fire and percolate into the underground waters. Earlier than 1970 (Feunge, 1951; Hamza, 1966), scientists had started recovering and using (Aoki, 1972; Kheoh, 1987; Kokama, 1980; Ong, 1983; Pushpinde, 1981; Sakakura, 1980) spent clay in bleaching earths used to decolorize vegetable oils (Alzaharani and Daous, 2000).

The use and disposal of by-products and spent materials from vegetable oil processing in the USA was studied (Alhameed and Zaharani, 2002; Watson et al 1976; Svensson 1976). Regeneration of spent earths (Bahl, 1977) was studied with water at elevated temperature and pressure (Ong, 1983). Regeneration of spent bleaching clay by heat treatment (Shu-Chen, 2000) in a gas stream was performed in a thermo-gravimetric analysis system.

Spent earths were shown to contain 17 – 35% residual oil, metallic impurities and other organic compounds (Kalam, 1988; Svensson, 1976). The substantial oil content of spent earths makes it a suitable ingredient of poultry and animal feeds. The process of regeneration of spent bleaching earths has been reviewed by several authors (Bahl, 1977; (Boukerroui, 2009; Penninger, 1979; Watson, 1976).

2.12 Pillared and intercalated clays

Pillaring and intercalation of bentonites or montmorillonites has been shown to improve bleaching and diffusivity properties bleaching earths. Alumina-pillared clays were used to bleach sunflower-seed oils (Falaras et al, 2000).

2.13 Differential thermal analyses (DTA) studies

The different temperatures required to extract structural water from the lattices of clay minerals reflected the clay-mineral types. The amount of water released when clay was heated at high temperatures (Stab et al, 2006; Vaiga; 2007).

2.14 X-ray diffraction (XRD) studies

The clay powder x-ray diffraction patterns were recorded on photographic films as series of lines in the form of arcs concentric with the original x-ray beam and from the positions of lines, angles of diffraction and separation spacing of planes producing them.

XRD studies on montmorillonite (Mills et al, 1950) were used to show that clay exchanges oxygen in form of hydroxyl (SiOH) and (HOH) with hydration of oxygen to surface exposing

fresh oxygen previously in the body of the oxide. XRD hot stage analysis was also used to demonstrate the presence of a more thermally stable polymorph of the kaolinite (Ip et al, 2008)

2.16 Determination of surface area of powders

The adsorption of paraffins (Dandy, 1967) has been used to determine the surface area of a mesoporous sepiolite. Discussion of the use of Bruanuer Emmett-Teller (BET) plots of data obtained at relative pressures lower than 0.05, which yielded different straight lines down to relative pressures of 0.01 (Gregg and Sing 1964). They argued that these linear sections appear to be in the higher relative pressure ranges of isotherms made at 193 and 209.7K. He clarified that in the BET equation,

$$P/(P_0-P)X = 1/X_m C + P/P_0 (C-1)/X_m C \dots\dots 2.8$$

Where X is uptake of adsorbate at pressure, P, X_m is monolayer capacity; P₀ is saturated vapor pressure of adsorbate and C is a constant.

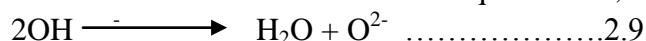
2.17 Sedimentation and hydrocycloning

Clay and clay minerals can be separated into different minerals following the principles of sedimentation and hydrocycloning (Kardy and Amer, 2007; Byarugaba et al, 2005). Different clay minerals have different wetting properties and so they have differing densities when wet. When the sedimentation mixture is left to stand, the different minerals will settle at different rates enabling separation of sediments in different layers. This way kaolinite, nontronite and other minerals can be separated and characterized.

2.18 Acid activation of clay

Activation of clay is a chemical or physicochemical treatment applied to clay to develop capacity to adsorb coloring matter and other impurities in vegetable, animal or petroleum oils (Lamar, 1951). The activity of clay denotes surface chemical and physicochemical reactivity leading to increase in surface area of solid (Gregg, 1992). The purpose of activating clay is for use as adsorbent for liquid, solid or gas or obtain high rate of reaction or dissolution. Activation of clay

aims at producing modifications on surface of material. Thermal dehydroxylation occurs at high temperature between 600 and 1000°C follows the chemical reaction shown below in equation 2.9,



Acid activation involves removal of exchangeable interlayer ions as well as dissolution of tetrahedral and octahedral ions from alumina and silica layers (Vanzuela-Diaz and Souza-Santos, 2001) and replaces them with hydrogen causing structural deformations. The mineral composition and texture of acid-leached clays changes (Franus et al, 2004; Rozie et al, 2010)

MATERIALS AND METHODS

3.1 Materials

Clay samples will be collected from the following places:

- a) Central Uganda; this will be at the following areas:
 - i. Kajansi in Wakiso District.
 - ii. Seeta and Lwanda in Mukono District.
 - iii. Kawuku and Nakawa in Kampala District.
- b) Eastern Uganda; this will be at the following areas:
 - i. Umatengah, Chodah, Kumi in Kumi District.
 - ii. Mutufu, Budadiri in Sironko District.
 - iii. Siron, Chelel in Kapchorwa District.

Samples of clays will be collected from Central and Eastern Uganda regions suspected to contain mainly kaolinite. The volcanic region of Mont Elgon near Kapchorwa and Sironko will be sampled for smectites because of past volcanism in the area. Sunflower seed, cotton seeds and date palms will be collected from Kampala's Balikuddembe market, Jinja oil mills and BIDCO, Jinja, pressed to collect the different oils.

3.1.1 Preparation of clay samples

Raw samples of clays will be separately soaked in distilled water, sieved to pass through a mesh of

5.3×10^{-4} m diameter, dried at 105°C and ground to powder using a rolling mill. The clay powders will be stored for future use.

3.2 Methods

The procedures to be used in the research are outlined herein as subsections.

The first objective will be achieved from the following methods;

3.2.1 Chemical, mineralogical and structural analyses

The chemical analyses will be determined by decomposition using sodium carbonate fusion method (Hutchinson, 1974) in platinum crucibles. Silica will be determined by gravimetry and the other elements were analysed using the Perkin – Elmer 3030 model Atomic absorption spectrometer after dissolution of the sample in the hydrofluoric acid– perchloric acid digestion mixture.

3.2.2 X-ray diffraction analyses

The mineralogy of clays will be determined using X-ray Powder diffraction (Philips diffractometer with PW1710 control unit operating at 40kV and 30mA using the Ni-filtered Cu K_α radiation). The diffractograms will be automatically matched with JCPDS-cards in the computerized XRD CD-rom. Bulk mineralogy will be studied with randomly oriented air dried samples (Reynolds, 1989).

3.2.3 Infrared spectroscopic studies

The clay powder (0.003 g) will be mixed with potassium bromide (0.1g) ground to powder, pressed into discs. The infrared spectra will be run using the KBr discs using B10RD FT540 Fourier Transform IR spectrometer in the frequency range of $3700 - 400 \text{ cm}^{-1}$ (Russell, 1979).

3.2.4 Differential thermal Analysis (DTA)

Clay sample (0.5 g) and thermally inert reference sample, calcined alumina, will be placed in cavities of a nickel block and heated to 1100°C at the rate of 12°C per minute. The changes in mass and temperature of the sample in comparison to

the reference will be automatically recorded on a DTA graph. DTA studies will be conducted with Differential Thermal Analyzer.

3.2.5 Determination of clay pH

Weighed clay powder (1.0 g) will be placed in a beaker; water (20.0 cm³) will be added. The mixture will be stirred continuously and allowed to equilibrate for 30 minutes at 25°C. The pH-meter will be used to read the pH of the solutions.

3.2.6 Determination of surface areas of powders

Clay powder (0.1 g) will be out-gassed at -170°C by lowering the pressure to 0.01 cm of oil manometer. At the end of the evacuation period, the vacuum line will be closed off and nitrogen from cylinder pumped intermittently in the manifold containing clay powders. Sufficient time will be allowed after each pressure increase to ensure that the system attained equilibrium. The weight of nitrogen adsorbed on the clay will be determined by deflection of silica spring balances.

3.3 Calcining and bleaching properties

Clay samples and de-oiled spent clay (200.0 g) will be placed in a furnace operated at the temperatures ranging from 450-500°C for two hours.

3.3.1 The Leaching of clays

Clay powder (100.0 g) will be mixed with acid of appropriate concentration (500.0 cm³) in a flask. The mixture will be heated at 105°C for 4 hours; then cooled and filtered. The residue will be washed to neutrality with distilled water; then dried at 105°C in the thermo-stated oven. The dried leached powders will be labeled and stored for future use (Nwabanne and Ekwu, 2013).

3.3.2 Degumming of vegetable oils

Crude oil (100.0 g) will be placed in a flask, 85% phosphoric acid (1.0 g) added, the mixture heated at 90°C while stirring at 900 revolutions per

minute for 10 minutes under nitrogen blanket. The oil will be filtered under nitrogen.

3.3.3 Neutralization of oil

Mixture of the degummed oil (200.0 g) and 0.1M sodium hydroxide (10.0 cm³) will be placed in 250 cm³ Pyrex glass flasks, fitted with a magnetic stirrer. The mixture will be stirred vigorously for 10 minutes at room temperature and filtered.

3.3.4 Bleaching of the vegetable oil

Mixture of the degummed, neutralized oil (200.0 g) and appropriate clay powders (5.0 g) will be placed in 250 cm³ Pyrex glass flasks, fitted with a magnetic stirrer. The flask will be immersed in a thermo-stated iso-electric mantle at temperatures, 40, 50, 60, 70, 80 and 90°C. The mixture will be heated while stirring continuously for a further two hours at the set temperature under high vacuum. The hot oil and clay mixture will be filtered in nitrogen atmosphere and tested by measuring its absorbance, transmittance, refractive index, turbidity and characteristics like iodine number, acid value, and free fatty acid values.

3.3.5 Bleaching oils with recycling spent clay

The mixture of degummed neutralized oil (200.0 g) will be mixed and heated with calcined, acid-leached spent bleaching clays (5.0 g) at various temperatures ranging from 40 to 130°C for two hours under nitrogen blanket with continuous stirring. The mixture will be filtered and the bleached oil will be tested for clarity by measuring its absorbance at 550nm.

The third objective will be achieved from the following methods;

3.4 Analysis of bleached oils

The bleached oil will be analyzed for parameters such as iron and copper content, peroxide value, free fatty acids value, iodine number, refractive index, transmittance and turbidity to find out whether it will be suitable for human use.

3.4.1 Iron and copper content

The bleached oil (0.2 g) will be digested in the perchloric-nitric-hydrofluoric acid mixture (3.0 cm³) then made up to 25.0 cm³ with distilled water. The absorbance of copper will be determined at 580 nm using the atomic absorption spectrophotometer Shimadzu-AA-6200.

Similarly, iron will be determined at 478 nm when ammonium thiocyanate solution will be added to complex the ions.

3.4.2 Peroxide value (Pv)

Bleached oil (2.0 g) will be placed in a 250 cm³ flask, ethanoic acid (30.0 cm³) added together with chloroform 9.1:5.0) ratio. The mixture will be swirled, water (50.0 cm³) added and 0.5 mol of standard potassium iodide, followed by two drops of starch indicator and adding water (100.0 cm³). The mixture will be titrated with 0.01M sodium thiosulphate to discharge the blue colour.

3.4.3 Free fatty acid value (AOAC 94048)

Bleached oil (2.0 g) will be placed in flask; butan-1-ol (10.0 cm³) added; and the mixture titrated with ethanolic 0.5M potassium hydroxide solution to the faint pink phenolphthalein end point.

3.4.4 Iodine number

Resublimed iodine (1.3 g) will be dissolved in ethanoic acid (100.0 cm³) and slight excess chlorine bubbled through it. Oil or fat (0.1 g) will be placed in the 250cm³ volumetric flask containing chloroform (10.0 cm³) excess 0.1M iodine (15.0 cm³) will be added, stirred and mixture boiled, then cooled. The amount of unreacted iodine will be determined by back titration with standard thiosulphate solution.

3.4.5 Refractive index of eluted oils

Bleached oil (3 drops) will be dropped on surface of lower prism using Abbe's refractometer (A. Kruss model). Prisms will be closed and mirror adjusted until it will give the sharpest reading while the constant temperature water will be running through. (AOACS method 921.08c).

3.4.6 Transmittance of oil

The transmittance of bleached oil will be determined using an ultra violet – visible spectrophotometer. The transmittance and absorbance of the unbleached and bleached oils at 550 nm will be determined for each oil sample.

3.4.7 Turbidity of oil

The turbidity of eluted oil portions will be determined and recorded using a Jenway turbidometer (model 6035) in the wavelength range of 200 – 900 nm.

3.4.8 Determination of acid value

The test portion (0.1 g) will be placed in a reaction flask and dissolved in 1:1 (v/v) mixture of 95% ethanol and ethoxyethane (150 cm³). The solution will be titrated with standard 0.1M ethanolic potassium hydroxide solution using phenolphthalein indicator. The determinations will be duplicated for each batch of bleached and unbleached oils. The acid value, AV will be calculated using the following formula:

$$AV = \frac{56.1 \times V \times T}{M}$$

Where V is value of standardised potassium hydroxide solution used; T is the molarity of the standardized potassium hydroxide solution used; and M is mass in grammes of the oil portion taken.

3.5 Determination of surface acidity

Activated clay powder (0.2 g) will be weighed and placed in a flask. A 1.0 x 10⁻²M solution of 1-aminobutane in isohexane (20.0 cm³) will be added. The flask will be stoppered and shaken vigorously for 20 hours using an electric shaker at room temperature. The equilibrium mixture will be filtered and portions of the filtrate (5.0 cm³) will be pipetted into a clean flask, extracted with water (5.0 cm³); methyl orange indicator (2 drops) added and the mixture titrated with 0.004M hydrochloric acid to orange endpoint.

For each clay sample, four replicate determinations will be made corresponding to 0, 5, 10 and 20% acid in the leaching medium, respectively. Each clay type will be activated by

heating at different temperatures in the range between 40 and 80° C at intervals of 10° C from each temperature used.

The fifth objective will be achieved from the following method.

3.6 Isolation and characterization of smectites

The ground, sieved clay powders (10.0 g) from Chelel, Siron, Mutufu and Budadiri will be suspended in distilled water (500.0 cm³) at room temperature (25°C). The suspension will be continually stirred by a magnetic stirrer for two hours and then left to stand. The supernatant solution will be allowed to stand for 20 minutes and the solution will be siphoned out. The remaining suspension will be made up to 500.0 cm³ with more distilled water; stirred for two hours and left to stand again. The solid phases formed will be collected, dried, ground to powder and stored separately.

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APPENDICES

APPENDIX 1: Time framework

Acti vity	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D	J	F	M	A	M	J	J	A	S	
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5						█	█	█	█	█	█	█	█	█	█	█	█	█	█	█	█	█	█	█	█	█	█	█	█	█	█	█	█	█	█	█	█
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Description of activity

- 1 Literature review and proposal writing
- 2 Registration
- 3 Selection of sampling sites
- 4 Sampling of clays from Central and Eastern Uganda.
- 5 Sample preparation for sieving
- 6 Acid-leaching of sieved samples, drying and washing to neutrality
- 7 Bleaching vegetable oils using earths from different areas in Uganda.
- 8 Determining surface properties of earths used to bleach
- 9 Determining qualities of bleached oils
- 10 Recycling spent bleaching earths from the study.
- 11 Data analysis.
- 12 Thesis write up and submission

APPENDIX 2: Budget

Item	2012-2013	2013-2014	2014-2015
University Fees			
Tuition fees	1,800,000	1,800,000	1,800,000
Registration fees	12,000		
Examination fees	180,000	180,000	180,000
Administration fees	120,000	120,000	120,000
Laboratory fees	200,000	200,000	200,000
SUB TOTAL	2,312,000	2,300,000	2,300,000
Lab supplies and analysis			
Elemental analyses	500,000		
Solvents and reagents	4,000,000		
Glass ware	1,800,000		
Sampling bags	750,000		
XRDS	3,500,000		
SUB TOTAL	10,550,000		
Field requirements			
Car hire	1,920,000	1,920,000	
Accommodation	1,440,000	1,440,000	
Technical support during sampling	2,628,000	2,628,000	
Fuel for car	540,000	540,000	
Local travel expenses	1,800,000	1,800,000	
SUB TOTAL	8,328,000	8,328,000	
Office supplies/Literature search			
Stationary	330,000	330,000	330,000
Computer cartridges	800,000	800,000	800,000
Purchase of books	780,000		
Subscription to journals	450,000		
Literature searches/Internet	600,000	600,000	600,000
SUB TOTAL	2,960,000	1,730,000	1,730,000
Training courses/workshops	1,700,000	700,000	350,000
Student's stipend, \$ 200 per month	4,200,000	4,200,000	4,200,000
GRAND TOTAL	30,050,000	17,258,000	8,580,000