

2.1 sampling of clays

Soil samples were collected from five locations namely:

1. 5kg of Nile clays were collected from River Nile West Shendi.
2. 5kg of Nile clays were collected from River Nile East Shendi.
3. 5kg of Blue Nile clays were collected from Blue Nile from Khartoum.
4. 5kg of Blue Nile clays were collected from Blue Nile from Soba.

The outcrops range in thickness from a maximum of 6m to less than 1m. The examination of vertical sections showed different parts of randomly consisting of black soil and clay materials.

5. 5kg of Jurdiga were taken from Timman Shops which were collected from Elhasania Mountain.

2.2 Equipment

- Beakers.
- Desiccator.
- Platinum crucible.
- Muffle furnace.
- Analytical balance.
- Volumetric flasks.
- Titration flasks.
- Stirrers.
- Pipette.
- Burette.
- Oven.
- Water bath.
- Hydrometer.
- Mechanical shaker.

- Sievers.
- Flame photometer, model: AFP 100Flame photometer Biotech Engineering Management Company Ltd India.
- X-ray fluorescence, model: PANalytical. Geological Research of Sudan.
- X-ray diffraction, Central Petroleum Laboratories (CPL).

It is X Pert PRO console PW 3040/60 set of 20°C and 400 atm. The instrument was made in South Africa by Swedish company.

2.3 Materials and Reagents:

Distilled water , KOH ,NaOH ,Na₂CO₃ ,NaHCO₃ , Na₂NH₄HPO₄, NH₄Cl, NH₄OH , EDTA , Methyl Red , HCl, H₂SO₄ ,HNO₃ , HClO₃ , K₂Cr₂O₇ , Zn metal , H₃PO₄ , K₂CO₃ , (NH₄)₂ C₂O₄ , HF.

2.4 Preparation of solutions:

Different solutions were prepared:

EDTA Solution:

EDTA solution “ sodium salt of ethylenediamine tetra acetic acid , it is a white mesh crystalline powder ,easily soluble in water and its chemical formula is Na₂C₁₀H₁₄N₂.2H₂O having a molecular weight of 372.25.

Preparation of 0.01 M EDTA: 3.7225 g were weighed and dissolved in a beaker and then transferred quantitatively to a 1000 cm³ volumetric flask and filled to the mark with distilled water .

Preparation of standard Ca²⁺ solution:

1.000 g anhydrous calcium carbonate CaCO₃ powder was weighed into a 500 cm³ Erlenmeyer flask , then 1+1 HCl were added until all CaCO₃ had

dissolved .200 cm³ of distilled water were added and boiled for a few minutes to expel CO₂. Cooled, added a few drops of methyl red indicator adjusted to the intermediate orange color by adding 1+1 HCl. Then transferred quantitatively to a 1000cm³volumetric flask and filled to the mark with distilled water, that standard solution was equivalent to 1.00 mg CaCO₃ per 1.00 cm³.

Preparation of KOH and NaOH:

56.11 g of KOH were weighed in 100 cm³ beaker and then dissolved in distilled water and transferred to a volumetric flask and completed to 1000cm³ to give a solution of 1 M and consequently other solution of 0.01 M , 0.02 M , 0.05 M , 0.07 , 0.09 , 0.1 M , 0.2 M were prepared and by the same procedure 0.01 ,0.02 , 0.05 , 0.07 , 0.09 , 0.1 M , and 0.2 M NaOH were prepared .

Preparation of buffer solution of pH =10 (NH₄Cl,NH₄OH):

To obtain a buffer solution of pH = 10 , NH₄OH , NH₄Cl were used, 142 cm³ of NH₄OH were transferred in 250 cm³ volumetric flask and then 7.5 g of NH₄Cl were weighed in a beaker and dissolved in a distilled water and transferred to the same volumetric flask and completed to 250 cm³ to give a buffer solution of pH = 10.

Preparation of Erichrome black T indicator (EBT):

3.00 g of EBT were weighed in a weighing bottle and transferred to a small beaker; then15 cm³ of triethanolamine and 5 cm³ of ethanol were added and then transferred to the indicator bottle.

2.5 Preparations of clays:

Each of samples were ground into fine – powder particles dried , and kept in clean dry sample bottle for subsequent analysis.

Analysis of tap water before modification:

50 cm³ of tap water were taken in 250 cm³ conical flask and then 5 cm³ of buffer solution of pH =10 were added with two drops of EBT indicator , this solution was titrated against 0.01 M EDTA , until the pink color of the solution was changed to blue indicating the end point of titration . From the volume of EDTA the hardness of tap water was calculated. The results are shown in table (4) on page (39).

Modification of clays and hardness removal:

Modification by standard KOH:

50 g of dried ground Nile clay were weighed and then treated with 50 cm³ of 0.1 M solution of KOH , then the contents were heated using heating mantle and boiled for half an hour with frequent stirring , then allowed to cool , and filtered . The residue was transferred to a desiccator after well washed with distilled water and dried for further two hours, and then the clay was transferred to a bottle for water hardness treatment.

The dried clay was mixed with 150 g sand (or gravel or glass beads), then the mixture was transferred to a funnel containing filter paper for water filtration.

The tap water was used to estimated hardness by passing it through the funnel then were left overnight .50 cm³ of filtered water by West Nile clay was transferred to a 250 cm³ conical flask, 5 cm³ of buffer solution of pH = 10 and 2 drops of EBT indicator were added to the solution and then

titrated against 0.01 M EDTA until the end point by changing the color of solution from wine red to blue , the hardness of water was calculated .The method was repeated for clays treated with 0.01 ,0.02 , 0.05 , 0.07 , 0.09 , and 0.2M KOH .The results are in table (6) on page (40).

The procedure of West Nile clay was repeated for other clays and the results are given in tables (8, 9 and 10) on pages (43, 45 and 47).

Procedure (B): Modification of clays using standard NaOH.

The same procedure (A) for standard KOH was used for standard NaOH treatment of clays .The results are given in tables (5,7,9,10 and 11) on Pages (40,41,45,47 and 49).

2-6 Hardness removal system:

This study was a pilot- experiment study. The treated samples were produced by the following treatment beds including modified clay and inert material (molecular sieve filter) the detail experimental setup of the study was illustrated in fig (1). The experiments ran through the separation of 5 beds by changing the treatment of the clay with different inert materials. Each bed was made from modified clay with inert material. The tap water was passing through. The raw tap water and effluents water of pre- and post experiment bed were measured for total hardness.

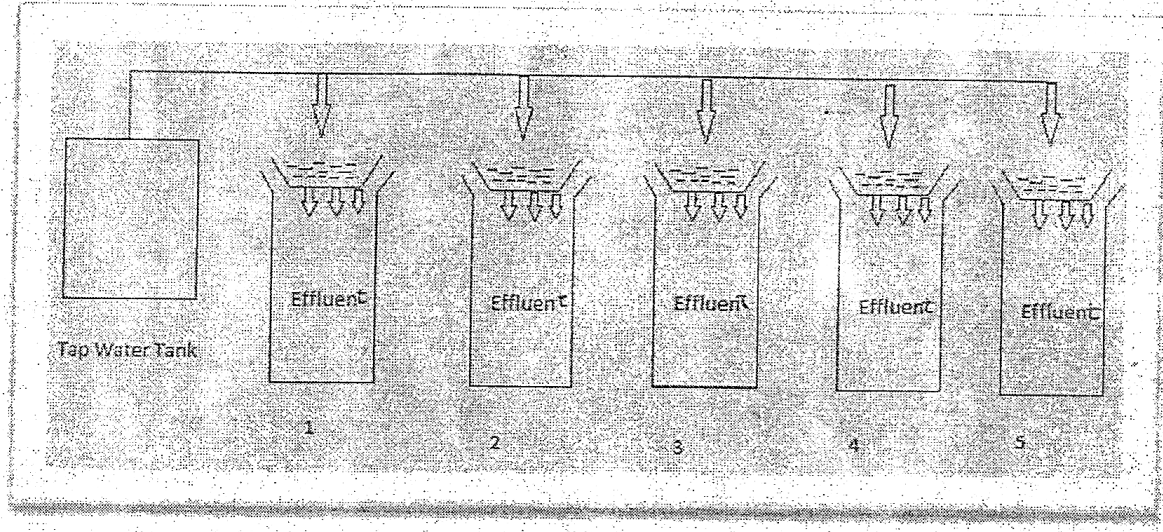


Fig (1) the experiment setup for continuous flow of bed with modified Sudan clays

Remark:

1. Is the bed which is containing 250g modified West Nile clay and 400g of sand (inert material filter).
2. Is the bed which is containing 250g modified East Nile clay and 400g of (gravel inert material filter).
3. Is the bed which is containing 250g modified Blue Nile clay and 400g of glass beads (inert material filter).
4. Is the bed which is containing 250g modified West Soba clay and 400g glass beads (inert material filter).
5. Is the bed which is containing 250g modified Jurdiga clay and 400g of peace of marbles (inert material filter).

2-7 Characterization of Clays:

Physical analysis: grain size distribution

100g of each powdered sample were separated through a stack of sieves by mechanical shaker. The amount in each sieve was weighed.

50g of weighed dried sample were added to 2.5g of sodium hexameta phosphate salt. Then the mixture was dissolved in distilled water and washed in 0.063mm sieve with distilled water after sieving of each samples, and the fraction >0.063 was treated using hydrometer analysis method.

The temperature in different periods through the experiment was taken, and then the results were given in figures (13, 14, 15 and 16) on pages (63, 64, 65 and 66).

Chemical analysis: clays were analyzed using standard methods of analysis gravimetric, volumetric and spectrophotometric procedures.

Loss on ignition

About 1.0000 g from each sample was accurately weighed in a clean weighed platinum crucible , the crucible was heated to 1000°C in an electric muffle furnace for one hour, the crucible was cooled in a desiccator and weighed ; the calcination was repeated to obtain constant weight.

The loss on ignition was determined as a percentage mass of the sample.

Determination of SiO₂:

5 g of fusion mixture (potassium and sodium carbonate) was added to each sample clay .Three quarters of this amount was mixed well by means of spatula. The remainder of the flux was then heated in the muffle furnace at 1000°C for 2 hours.

The crucible containing the fused silica was placed in the porcelain dish to remove the fusion mixture and then HCl was evaporated in the water bath. The silica was baked at 110° C for 2 hours and then the contents were moistened with dilute HCl and then filtered off. The filtrate was evaporated to dryness , the residue was heated at 110° C for 2 hours , and then the residue was treated with dilute hydrochloric acid and then filtered on a filter paper .The filtrate was placed into 500 cm³ volumetric flask and then completed to the mark with distilled water .The solution was used in all subsequent determinations of combined oxides, Ca⁺² , Mg⁺² , the washed precipitates were combined , ignited at about 1000° C to constant weight and then treated with 10 cm³ of hydrofluoric acid and 5 cm³ of concentrated H₂SO₄ to purify the silica .

Determination of Al₂O₃, Fe₂O₃ (combined oxides):

To 100 cm³ of the obtained solution after silica determination, 3 g of ammonium chloride crystal were added. The solution was heated and then two drops of methyl red indicator were added followed by dilute ammonia.

The mixture was heated to boiling in a water bath for 2 minutes and then filtered. The filter paper with residue was ignited to 1100C⁰ in a weighed crucible to constant weight; then Fe₂O₃ + Al₂O₃ percent by mass was obtained.

Determination of Fe₂O₃:

To 50 cm³ of the solution obtained after silica determination, zinc

(metal) was added and the mixture was heated in a water bath until the solution became colourless, 10 cm³ of phosphoric acid were added to the solution and 3 drops of diphenylamine sulphonic acid indicator were added and titrated with 0.016 M K₂Cr₂O₇ , the amount of Fe₂O₃ % by mass was obtained , and then Al₂O₃% by mass was calculated.

Determination of CaO:

To the combined filtrate after combined oxides determination, HCl was added to a weakly acidified medium, heated to boiling. 1.5 g of (NH₄)₂C₂O₄ were added and heated to boiling NH₄OH solution was added until the medium was basic and the precipitate was allowed to settle for 2 hours on water bath , then filtered . The residue was ignited at 1100° C to a constant weight in a weighed crucible, then cooled in a desiccator and weighed as CaO.

Determination of MgO:

To the subsequent filtrate, HCl was added to 10 cm³ of Na₂HPO₄ solution were added with stirring to allow complete precipitation overnight, and then filtered off. The precipitate was ignited in a weighed crucible at 1100° C, cooled in a desiccator and weighed as Mg₂P₂O₄. MgO was then calculated.

Then the results of chemical analysis were given in table (18) on page (67).

Determination of K₂O, Na₂O:

0.5 g each powdered sample were weighed in a Teflon dish, then moistened the sample with a few drops of water, 10 cm³ of HClO₄, 10 cm³ of HF and then 10 cm³ of HNO₃ were added to each sample.

The contents were evaporated in a sand bath to dryness then cooled, 30 cm³ of distilled water were added with 5 cm³ of HCl, and then placed in the sand bath until clear solution was obtained, then filtered in 500 cm³ volumetric flask. The standard solutions (1-6) were prepared for each Na⁺ and K⁺.

Standard 1 = 2 ppm, standard 2 = 4 ppm, standard 3 = 6 ppm, standard 4 = 8 ppm, standard 5 = 10 ppm, standard 6 = 12 ppm.

The results obtained of the flame photometer analysis were given in table (19) on page (67).

2-8Chemical analysis by x-ray fluorescence spectrometry (XRF):

Five powdered samples were prepared by melting 1.00g of each powdered sample using 10g flux (lithium tetra- and meta borates) 66:34, respectively. Fused beads were prepared by using an automatic bead instruments, the major elements constituents were determined. Pressed pellets for trace elements determinations were prepared by mixing 7g of each sample with 1.6g of binding wax through the computerized mini- mill grinding machine for one minute on 38 rpm speed, then the mixture was pressed in a standard aluminum cup using automatic pressing machine. Then the results were given in tables (20 and 21) on pages (69and 70)

2-9Mineralogical composition of samples by X- ray diffraction analysis (XRD):

The sample were disaggregated gently and carefully in a porcelain mortar using a spatula, a slight excess of powder was placed in the sample holder and pressed vertically into a smooth flat – surfaced powder cake using a microscope- mount flat leveling press. XRD patterns of soil were obtained on powder x-ray diffractometere .then the results were given in fig (17, 18, 19, 20 and 22) on pages (73-77).