UNIVERSIDADE D
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Characterization of Kaolin Deposits in Aileu Sub-district, Aileu District (Timor Leste)

Master in Geosciences

Specialization in Mineral Resources

Scientific advisors

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ABSTRACT

According to the published regional geology, there are two formations in the study area, namely the Aileu Formation and the Ainaro Gravel Formation. Based on geological studies on a scale of 1: 25,000, the lithology of the study area can be divided into four units, namely low alteration and mudstones, quaternary gravel units, kaolin units and alluvial deposits.

The mineralogical analysis shows that the analyzed samples are constituted mainly of illite/kaolinite and quartz, followed by muscovite and some goethite. The chemical analysis data from these samples reveal oxides, namely SiO_2, Al_2O_3, Fe_2O_3 and K_2O which are consistent with the identified mineral composition. Fe_2O_3 values are generally high, usually more than 1%, averaging between 2,6% to 3,2%, and the highest values (AL-20T) are higher at 5,9%. Grain size distribution leads to groups dominated by silty-clay, except for AL-31 which is dominantly clay. Technology testing, after drying and firing, shows that, for the kaolin samples, most of the total shrinkage occurs during the drying stage up to 110 °C and that it is reduced during the firing stages. Regarding the mechanical flexural strength, the parameter increases with the firing temperature, being the most significant increase in samples AL 17 and AL 31.

The analysis and measurement of the parameters took place at the “Centro Tecnológico da Cerâmica e do Vidro” (CTCV), Coimbra - Portugal.

The physical, chemical and technological characteristics of the Aileu's kaolin allow industrial use in the red-based structural ceramics segment, as a component of a mixture involving other types of clay (e.g. montmorillonite) and sand in order to optimize the ceramic paste. The amount of resources are significant.

Key words: Aileu formation (Timor), kaolin, technological tests, commercial uses, non-metallic resources

RESUMO

Com base na geologia regional, existem duas formações na área trabalhada, a Formação Aileu e a Formação das Areias de Ainaro.

Com base em estudos geológicos com uma escala de 1: 25.000, a litologia da área de estudo pode ser dividida em quatro unidades, e que são: argilitos de baixo e alto grau de alteração, cascalheiras do quarternário, unidades cauliníticas, e depósitos aluviais. A análise mineralógica mostra que as amostras são constituídas por ilite/caulinite, quartzo e, com menor representatividade, moscovite e goetite.

A partir dos dados das análises químicas, as amostras contêm óxidos, nomeadamente SiO_2, Al_2O_3, Fe_2O_3 e K_2O, o que é consistente com a composição mineral identificada. Os valores de Fe_2O_3 são geralmente altos, normalmente acima de 1%, com valores médios entre 2,6% e 3,2%, apresentando a amostra AL-20T o valor mais alto e que é superior a 5,9%.

A distribuição das dimensões dos grãos incluem-nas no campo dos siltes argilosos muito finos, exceto a AL-31 que se enquadra nas argilas.

Testes tecnológicos, após secagem e cozedura, comprovam que estas amostras cauliníticas possuem uma retração total essencialmente na fase de secagem a 110 ° C, sendo reduzida durante as etapas de cozedura ensaiadas.

Em relação à resistência mecânica à flexão, este parâmetro aumenta com a temperatura de cozedura, sendo o aumento mais significativo nas amostras AL 17 e AL 31.

Os métodos de análise e parâmetros laboratoriais obtidos foram realizados no Centro Tecnológico de Cerâmica e Vidro (CTCV), Coimbra - Portugal.

As características físicas, químicas e tecnológicas do caulino de Aileu permitem o uso industrial desta matéria prima no segmento da cerâmica estrutural de base vermelha, como componente de uma mistura envolvendo outro tipo de argilas (e.g. montmorilonite) e areia, para otimizar essa pasta cerâmica. Os recursos são significativos,

Palavras chave: Formação Aileu (Timor), caulino, ensaios tecnológicos, usos comerciais, recursos não metálicos.

Contents

LIST OF FIGURES

LIST OF TABLES

CHAPTER I. INTRODUCTION

I.1 Introduction

The country of Timor-Leste, being a very young country, must begin to evolve in order to achieve the goals of the independence for which it has fought for. The development will succeed if a balance between the availability of educated human resources and proper use of existing natural resources potential can be found.

Non-metallic minerals are the mineral commodities which are not included in the groups of the metal minerals, coal or other energy minerals. Non-metallic minerals are commonly referred to as non-metallic minerals or industrial excavation materials or group C excavated materials. Non-metallic minerals are easy to find and the businesses do not require large amounts of capital, complicated technology and long time for exploration, so they are suitable for increasing the people's economy.

Aileu municipality has been selected as the study area that has the potential in non-metallic mineral that can be used in the industry. Mineral resources that exist in study area such as kaolin, the availability of the kaolin itself, are controlled by geological processes like the hydrothermal, residual deposits, which are classified as primary deposits, and other occurrences such as sedimentary deposits, classified as secondary. The physical and chemical conditions under which kaolin forms are relatively low temperatures and pressures. The most common parent rocks are granites and rhyolites and the most common parent minerals are feldspars and muscovite. The physical and chemical properties of kaolin determines its ultimate utilization.

As it is known, some kaolin can be used as paper coating clays, some as filler clays in several industries, some is used for ceramics and refractories and some is used for special uses. So the purpose of this study is to identify, characterize and assess the possible use of kaolin from Aileu and surrounding area, as well as to determine the physical and chemical characteristics of the kaolin to assess the potential value for industrial materials.

I.2 Purposes and Objectives

I.2.1 Purpose

The ultimate purpose if to fulfill the requirements of the Master degree in Geosciences – branch Mineral Resources - of the Earth Sciences Department of the Faculty of Sciences and Technology of the University of Coimbra. This Master's dissertation is entitled "Characteristics of Kaolin in the Aileu and surrounding area" and it contributes for the understanding and utilization of this particular kaolin.

I.2.2 Objectives

- To identify the geological resources of non-metallic minerals (kaolin) of Timor Leste;
- To evaluate the characteristics of Kaolin in the Aileu district, in relation to the chemical, physical and technological properties;
- To evaluate the industrial applicability of kaolin from the Aileu area;
- To estimate the resources of the Aileu Kaolin.

I.3 Location of study area

Administratively the study area is located in the Aileu district which consists of one sub-district (Aileu sub-district) and 5 sucos (villages) (Suco Seloi, Suco Seloi Craik, Suco Hurairaco, Suco Sarin, no suco Aisirimou).

Geographically, the study area is located between the coordinates 125°30'0.03"E-125°34'8.48"E (longitude) and 8°42'.0"S - 8°44'.45"S (latitude), in an area of about 8 x 6 kilometers (Figure 1). Regarding the accesses to the study area it takes about one hour's driving by car or motorcycle to go from Dili to the study area (Aileu town). The road access from Dili to Aileu town is in good condition, although some parts of the road from Dili to Darelau are still under construction, and some parts are unpaved, but from Darelau to Aileu the road is already paved and in good conditions. But in the study area, there are some restrictions and some observation points can only be reached by a foot path or by walking on the river bed. The SW part of the

study area is quite difficult to access because of the thick vegetation and steep topographic features.

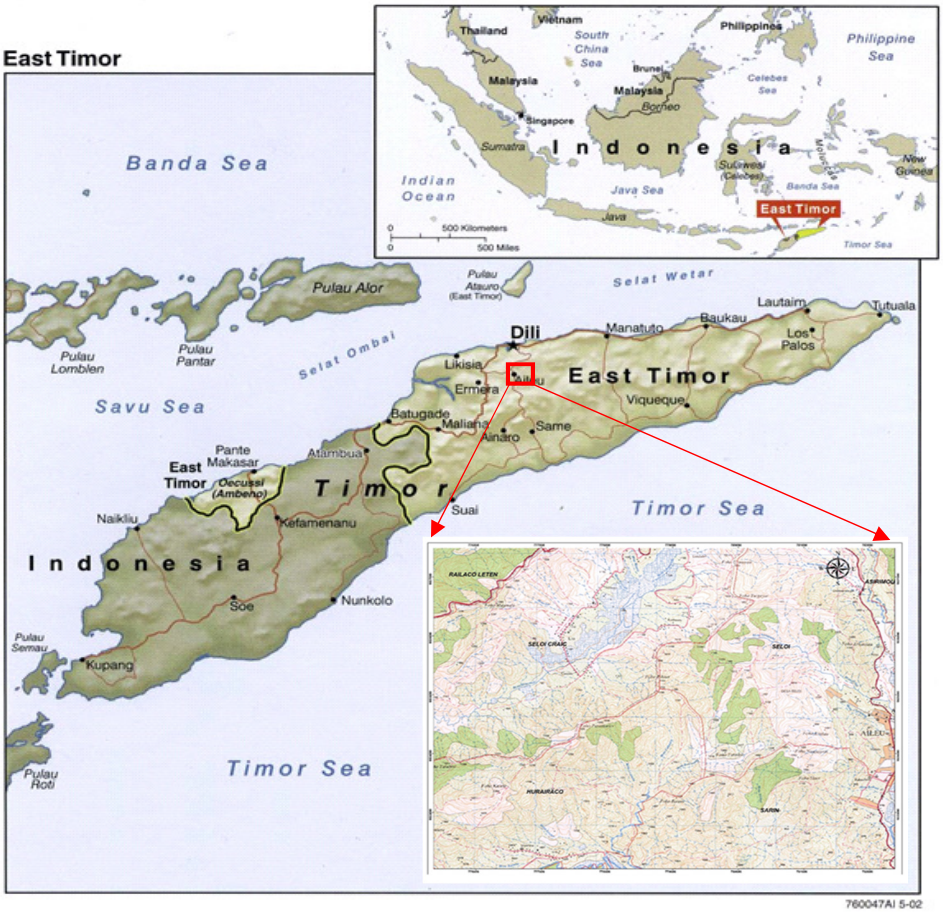

Figure I. 1 Location of the study area in the Aileu district – Timor Leste (Sourc: https://legacy.lib.utexas.edu/maps/east_timor.html)

CHAPTER II. GEOLOGY AND KAOLIN DEPOSITS IN STUDY AREA

II.1 Regional Geology

Based on regional geological maps, the geological of study area can be divided into two formations from younger to older age which are the Ainaro formation and the Aileu Formation (Audley Charles 1968). The Ainaro Formation is generally an alluvial terrace which consists of various fragments such as basalt, ultrabasic, limestone, and also chert sedimentary rocks with various grain sizes from boulder, gravel, pebble, sand down to silt. In addition, it shows a thin to thick layered sedimentary structure and also cross bedded. Ainaro formation is considered a quaternary alluvium deposited on the river bank with a thickness of approximately 15 meters. The Aileu formation occupies a large extent in the Timor territory (almost all of the northwest) and extends from around Manatuto (north of Laclo), bordered in the south by Aileu and it extends west to the area around Balibo and Batugade. It was presented by (Audley-Charles, 1968) as allochthone corresponding to the rocks of Permian in Timor-Leste (Figure II.1). Aileu formation consists of metamorphic rocks produced as a result of the collision of the northwestern part of the Australian Plate with the Banda Islands Arc. These collisions are estimated to occur in the Late Miocene and last until the present with a very young age in the world and form a very complex zone of metamorphism (Berry & McDougall, 1986). According to Prasetyadi & Harris (1996) metamorphic rocks from the Aileu formation are further divided into two parts, which are a meta-sediment (phyllite, schist, slate and marble) and a meta-igneous (metabasalt, metagabro and serpentinite). Metamorphism which occurs in the Aileu Formation is a metamorphism that occurs multiphase where the metamorphic rocks are re-experiencing metamorphism (Prasetyadi & Harris, 1996; Standley & Harris, 2009). The formation of the Aileu Formation metamorphic rocks consists of three stages (Prasetyadi & Harris, 1996). The first stage is the formation of metamorphic rock photoliths in a quiet epicontinent environment. Metamorphism progradation related to the rifting process occurs through penetrative deformation and metamorphism occurs in green schist facies. The second stage is metamorphism during the collision. Ductile deformation occurs, metamorphism occurs in moderate P-T conditions or on facies of green-amphibolite schist. The third

stage is the result of metamorphism dislocation and uplift of impacted rock masses. At this stage locally developed folds, extensional faults, and backthrusting happened with low degree alteration, minor recrystallization, and cooling. Based on Prasetyadi & Harris (1996), the environment continuously calm is reflected by metasedimentary rocks in the form of metapelit and interbreeding of thick marble in layers. Metamorphic rocks are formed in facies of metamorphic green sub-schist (Prasetyadi & Harris, 1996). Higher degree metamorphic rocks, in the Aileu Formation, formed locally due to mafic rock intrusion (Prasetyadi & Harris, 1996). Mafic rock intrusion is related to the occurrence of rifting by not finding fold deformation in schistosity. Metamorphic rock is formed in facies of green schist to amphibolite (Prasetyadi & Harris, 1996). Metamorphic rocks with folded schistosity are formed in metamorphic rocks higher degree (Prasetyadi & Harris, 1996). Steven Borger (2011-2012) conducted mapping in the Laclo area (Manatuto district) with a scale of 1: 50,000 and identified several rock units from old age to young age such as phylite and slate (Ppy), graphite, intercalation schist with sandstone (Psh), arenite, archose intercalation with phyllite and schist (Pss), gray marble (Pma), the Beheda formation which consists of marble (Nma) with an intercalated hornblende mafic gneiss layer with olive green gneiss, amphibolite (Nhm), coarse grained amphibolite (Nsb) which is part of the Fatucama suite and also in the Aileu complex there are also materials from Banda such as ultramafic gneiss rocks (Nlz) and serpentinite.

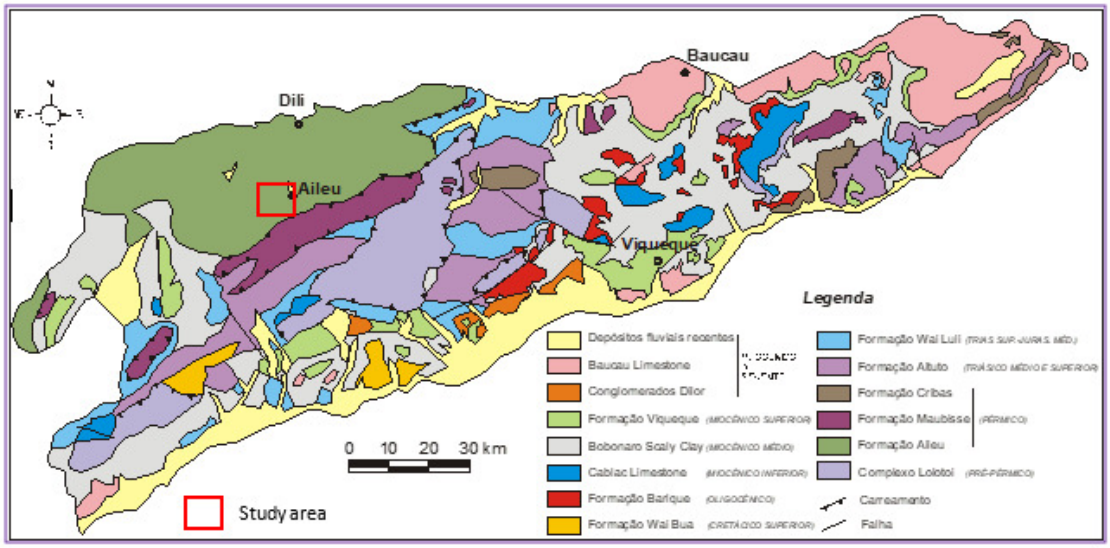

Figure II. 1 Simplified geological map of East Timor (adapted from Audley Charles, 1968)

II.2 Geology of study area

The preparation of the study area stratigraphy is based on the lithology concept developed in the stratigraphic arrangement covering the geological age, lithology characteristics and types of rock units observed in the study area. The naming of these stratigraphic units uses unofficial litho-stratigraphic names. Determination of the unit age is based on the correlation with the regional geological (Audley Charles 1968).

The stratigraphy of the study area consists of four rock units. The sequence of rock units from older to younger is Mudstone unit, kaolin unit, quaternary gravel units and alluvial deposition. Based on the type of surface weathering or alteration at the rock unit, the mudstone unit can divided into sub-units which are the mudstone low alteration units and the mudstone high alteration units.

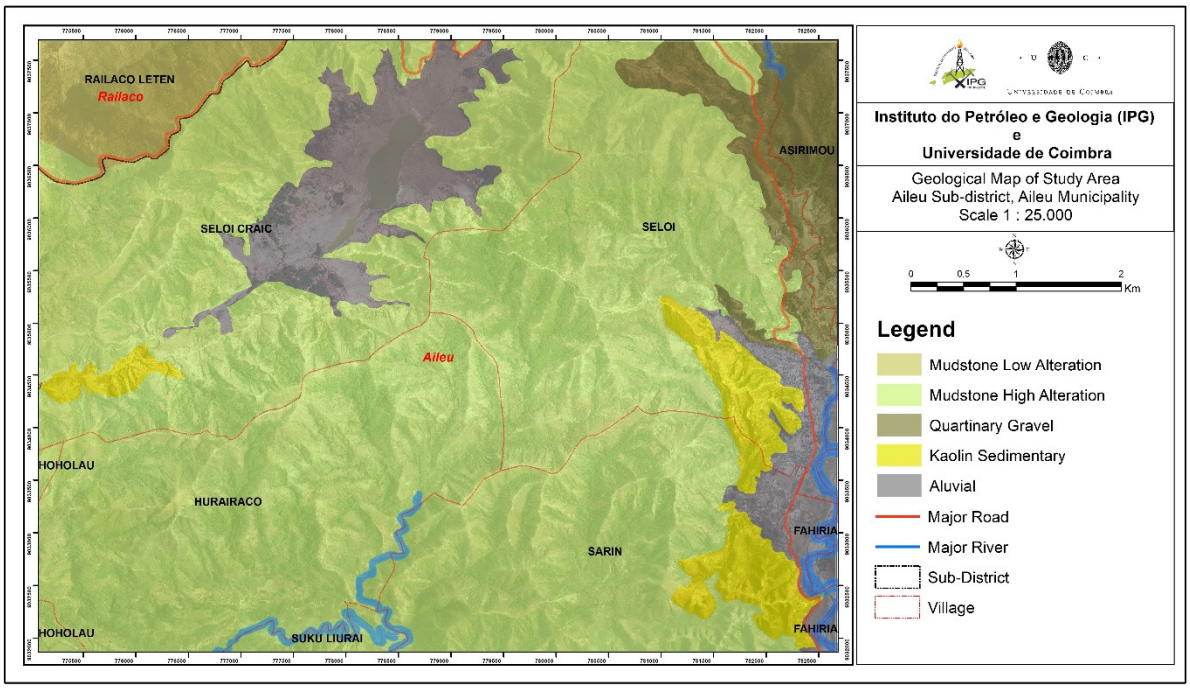

Figure II. 2 Map showing the rock unit distribution in study area, Aileu Municipality

1. Mudstone Low Alteration

The oldest unit that is found in the study area. This unit has an area of 1,8 km^2, 10% of the total area of study. Located in the north and west of the study area (Figure II.2). Based on observation in the field, this unit, in megascopic view, shows a variety

of gray, reddish black, fine-grained clastic rock, it is not well layered, and it contains more clay but fragments consisting of quartz and silt, the matrix consists of silt in a silicate cement. This unit breaks into blocky pieces.

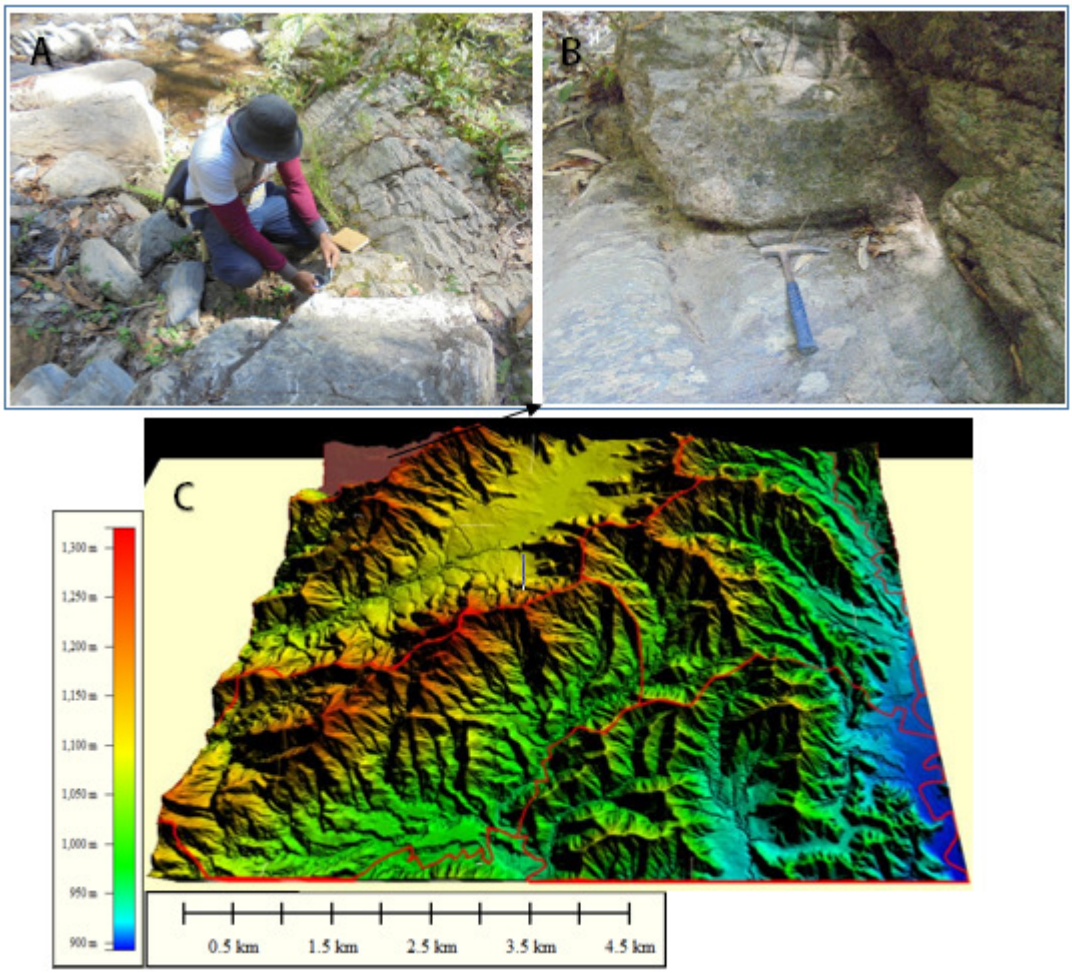

Figure II. 3 (A and B) Outcrop of Mudstone low alteration unit in the study area (C) 3D SRTM showing the location of this unit in study area at Railaco Leten Village (Arcgis 10.2).

2. Mudstone high alteration

The high alteration mudstone unit is the second unit found in the study area. This unit has an area of 33 km^2 of the study area. It spreads around all the study area dominating from the south-west to the north-east (Figure II. 4). This unit, in megascopic view, shows white, gray, yellowish white, reddish, thin layer, blocky structure and a texture: silt-clay, closed fabric. It is composted of quartz fragments, silt, and iron oxide. There is a quartz vein that is parallel to the bedding plane and also there is a cutting of bedding plane with a thickness of 1 cm-5 cm and there is

thermal fluid activity that fills these rocks through fractures and converts these rocks into kaolin with indications of white, red and yellowish white.

The texture shows foliation and it has a low metamorphosis and some indications of sedimentary structures such as lamination are still present.

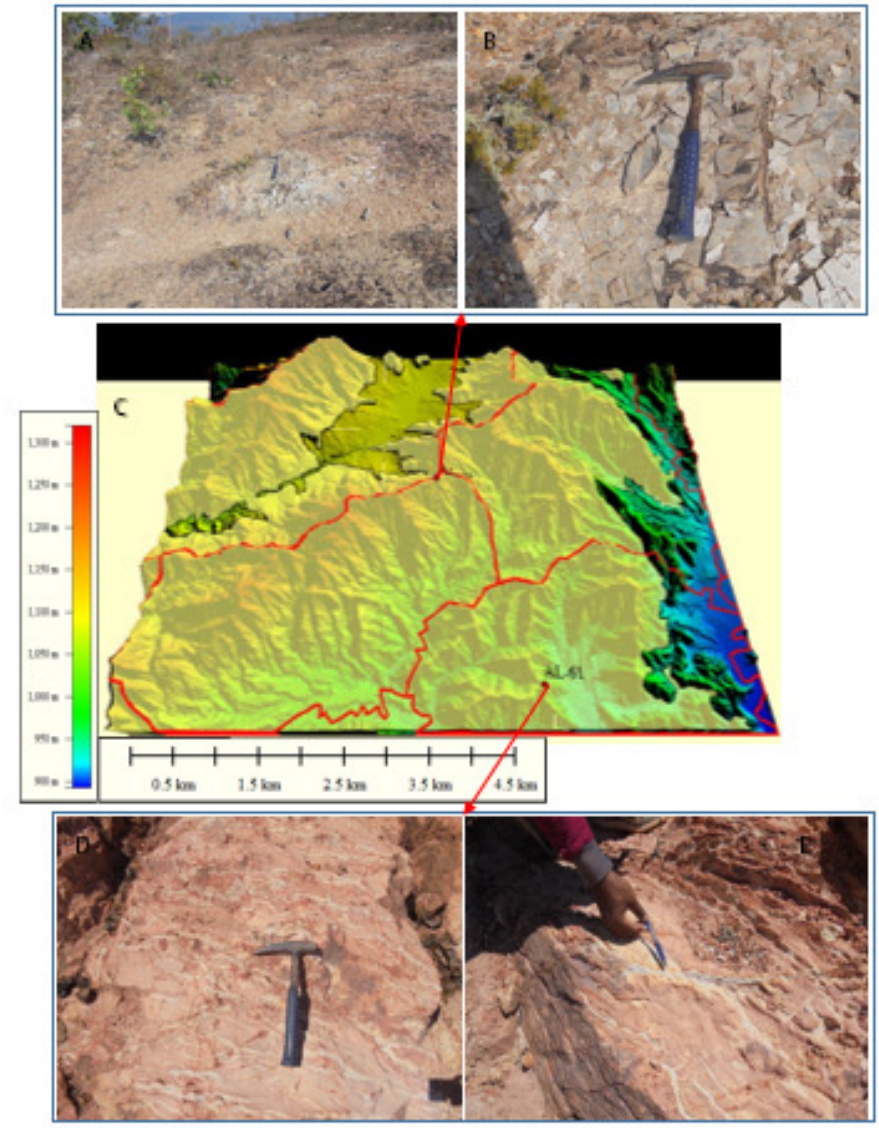

Figure II. 4 (A and B) Indication of mudstone outcrops showing blocky structure (C) 3D SRTM data to show morphology condition, location and distribution of this unit (Arcgis 10.2); (D and E) outcrop of this unit filled with hot liquids through fractures which are shown in white, yellow, reddish on each fracture. This is an indication of residual kaolin in the host rock.

3. Quaternary Gravel unit

This unit has an area of 2.4 km^2 of the total area of the study area. It spreads from the eastern part to south-eastern of study area (Figure II. 5). There are two possibilities for the formation of this unit in study area: the first is that it can occur due to morphological factors, slope, which caused the rock material above (initially strong) exposed to the sun, rain and weather so that the strength of the rock was lost and it was transported to the bottom and deposited in the anticline wings or flat areas that have an height of approximately 50 m. The second factor is that it occurs because it was caused by water that transported the material which settled in the river. The river terraces, with a maximum apparent thickness of about 20 m, belong to the Quaternary Gravels unit. They now lie about 980m above sea level and are stranded above the present river bed. These ancient terraces are composed of typical alluvium: slate fragments, mudstone fragments and quartz boulders and pebble-size material, sands, silts and clays. This gravel, in megascopic view, shows color variations such as white, gray, yellowish white, reddish, texture: gravel - clay, sorted bed, open fabric. From the position of the terraces above sea level, it can be inferred that they are of under post-Pliocene age or considered as quaternary gravel.

Mineral composition: fragments consist of quartz and mudstone, matrix consist of quartz, silt, iron oxide. Seen from the angular shape of the fragments (quartz, mudstone), it can be concluded that the process of material transport was not far from the source. It can be said that the gravel formation process was possibly formed or caused by morphology conditions, slope, and structure with transport by water and material delivered from the upper parts to the lower parts where they deposited. There are several evidences from the randomly spreading of the gravel near the river plain area which forms the river terrace and it always intercalates with kaolin (thin thickness) and where the kaolin is dominant it is considered part of the kaolin unit.

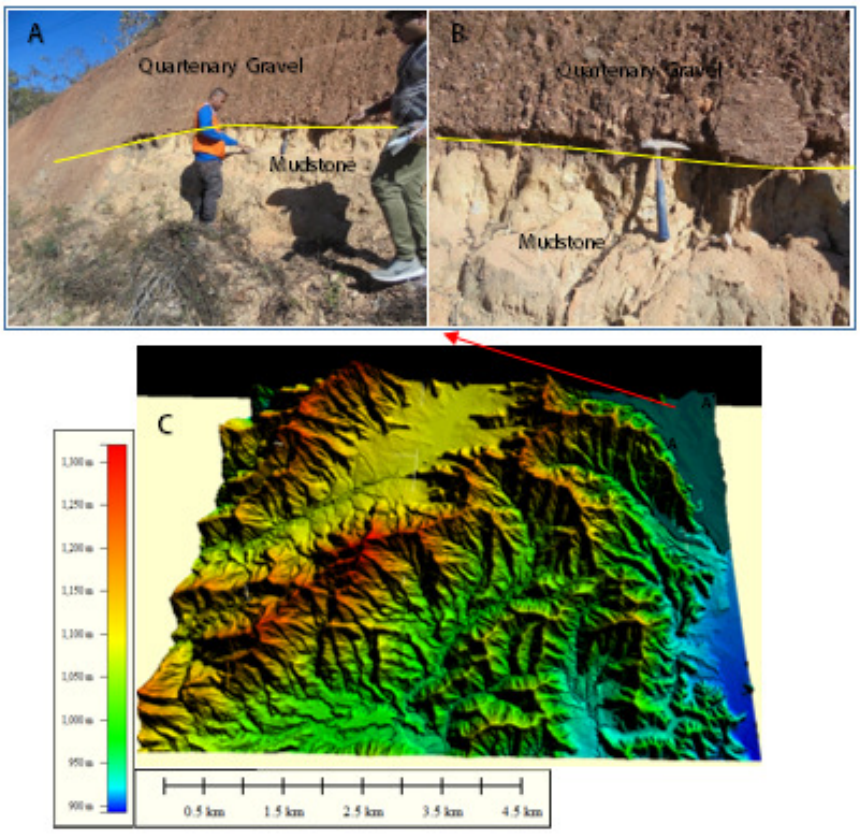

Figure II.5 (A and B) Gravel Outcrop (TOP) with mudstone (BOTTOM), thickness layer of gravel ±5 m and thickness of mudstone ±2 m
(C) 3D SRTM data show location distribution of gravel and morphology condition (ArcGis 10.2).

4. Kaolin unit

Kaolin unit considered as sedimentary kaolin: this unit in the study area is located in the eastern part close to the Aileu town around the Kabasfatin area, Sarin village and others located in Satu area, and Seloi Craik village in the west of the study area. There are four different characteristics showing varied colors from light-colored, exhibiting white, yellowish or greyish colors, with yellow to brownish areas locally, and sometimes with brown ferruginous specks.

5. Alluvial deposits

The Alluvial Deposit Unit covers an area of 4,8 km^2 of the study area (Figure II.6 C). This unit is found in the northern part and spreads in the east-west direction of the study area, precisely in Suco Seloi Craik. The thickness of this unit is estimated at ± 5-10 meters. This unit was formed due to the deposition of various materials such as alluvial and colluvial which also came from various origins such as rocks from the Aileu formation and gravel quaternary and surrounding area. Alluvial soils are classified as young soils, which are formed from fine sediments in streams from slope conditions so that the above material falls and is deposited in lowlands (Figure II.6 A,B). Nutrients are relatively high and there is a lake, especially in the Seloi Craik area which is now used as a tourist place.

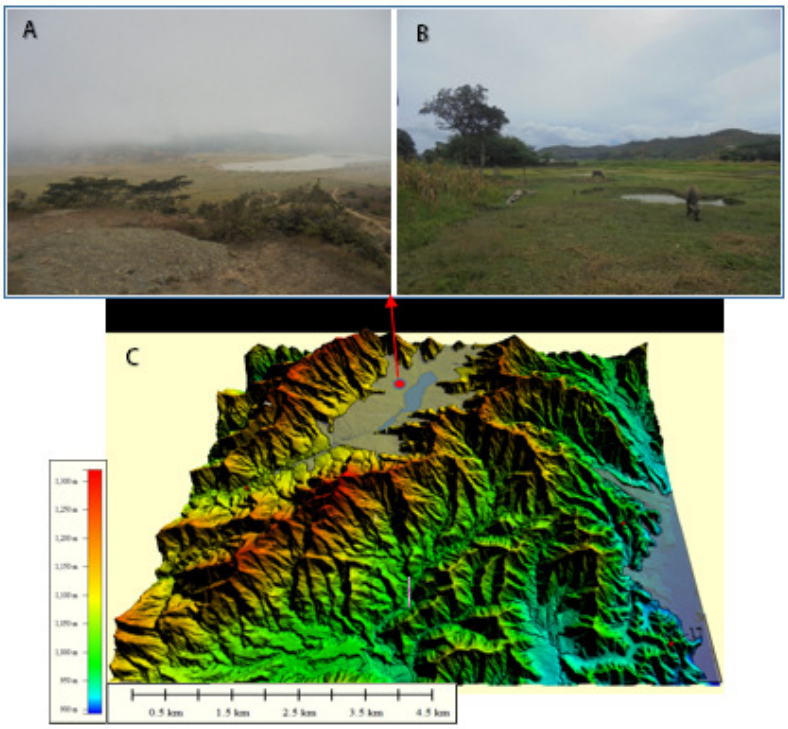

Figure II. 6 A. Alluvial unit from remote photo, at Seloi Craik village

B. Alluvial unit from close-up photo, Seloi Craik (Photo IPG, 2019)

C. 3D view by SRTM data, this unit is located in two villages namely Seloi Craik village and Seloi village. (Arcgis.10.2).

II.3 Kaolin Deposits in study area

Kaolin in the study area is a rock mass composed of silt-clay material and it is generally white, and there are also a number of colors that affect the color of kaolin such as yellow, red and gray which are influenced by oxides and organic material in the surrounding environment.

Kaolin belongs to the clay mineral group with a hardness of 2-2,5 an is plastic. Based on genesis studies, kaolin deposits occur through two processes, namely primary and secondary sedimentation processes. The primary sedimentation processes occur through thermal fluid activity (meteoric water) and weathering processes. The secondary deposits occur through weathering, transportation and sedimentation processes in low areas.

In the hydrothermal process, kaolin is generated from the interaction between meteoric water (hydrothermal solution) with mudstone parent rock and with rocks containing potassium aluminum silica and feldspar minerals. Quartz veins are evidences in the field of the presence of hydrothermal activity and thermal fluid activity such as meteoric water which causes discoloration in weak places, such as fractures, or through spaces between rock grains. Based on this, kaolin deposits formed by the hydrothermal process are often found in cracks, and in permeable rock layers.

Conditions on the field show intensive weathering that occur on the surface or very close to the surface of the ground, showing a wider spread but in small amounts, and as we move towards the interior, the kaolin increasingly disappears. Evidences on the surface of the original rock, shows that it has experienced weathering and this is shown through the weathered nature, red, yellow colors.

II.3.1 Location and Sampling of Kaolin sedimentary

From the different physical characteristics such as the color of kaolin, three samples (10 kg each sample) were collected at each one of the different locations representative of each type (color) of kaolin. Then the samples were sent to the laboratory for chemical analysis, mineralogy, and technology testing in the laboratory to determine the character and quality of each.

The code AL-17 sample represents the white to white-gray kaolin located at the eastern part with an expression of about 0,8 km^2 of the study area. The deposition of this kaolin units is always intercalated with gravel (Figure II.7). The forming strata are tabular, with a thickness of layering generally between 10 cm and 2,5 m, and the total maximum thickness of the outcrop is approximately 50 m.

The code AL-20 sample represents the yellowish (TOP part of the outcrop) and the reddish (BOTTOM part of the outcrop) kaolin, this area is located in the eastern part with an approximate area ± 0,9 km^2 in study area. The deposition of this kaolin (Figure II.7) forms strata that are tabular, with a thickness of beds of approximately 3m in the bottom part and 2 m in the top. The altitude is 960 m above sea level.

The code AL-31 sample represents white kaolin; this area is located in the western part with an approximate area of 0,3 km^2 in the study area. The kaolin in this location consists of white kaolin that intercalates with gravel. In this outcrop, the estimated thickness of the gravel layer is about 1 m and the estimated thickness of the kaolin layer is around 2m. In the morphological conditions seen the kaolin spreads upstream topographically.

From the results of the field observations, the characteristics of the kaolin outcrops, as observed from the color of kaolin, show different mineral and chemical compositions. Weather, normal fluid activity and thermal fluid caused the old components to evolve to new compounds and produce colors that are varied in the study area. These colors are influenced mainly by three pigments: the brown or black colors are usually influenced by organic content, while the red and yellow colors indicate the presence of oxidized iron oxide and the white color indicate aluminum silica and salt.

The presence of water also influences the color of kaolin by affecting the rate of oxidation. Kaolin which has a higher water content will have little air especially oxygen. Kaolin which tends to dry and because it is richer in oxygen elements will generally be red caused by oxidation. The following is a combination of 3D images from SRTM data and field data to show the morphological conditions and location of the three kaolin areas sampled. These samples were brought to the laboratory for chemical analysis, mineralogy, and technology testing to determine each characteristic and quality of each color difference (Figure II.7)

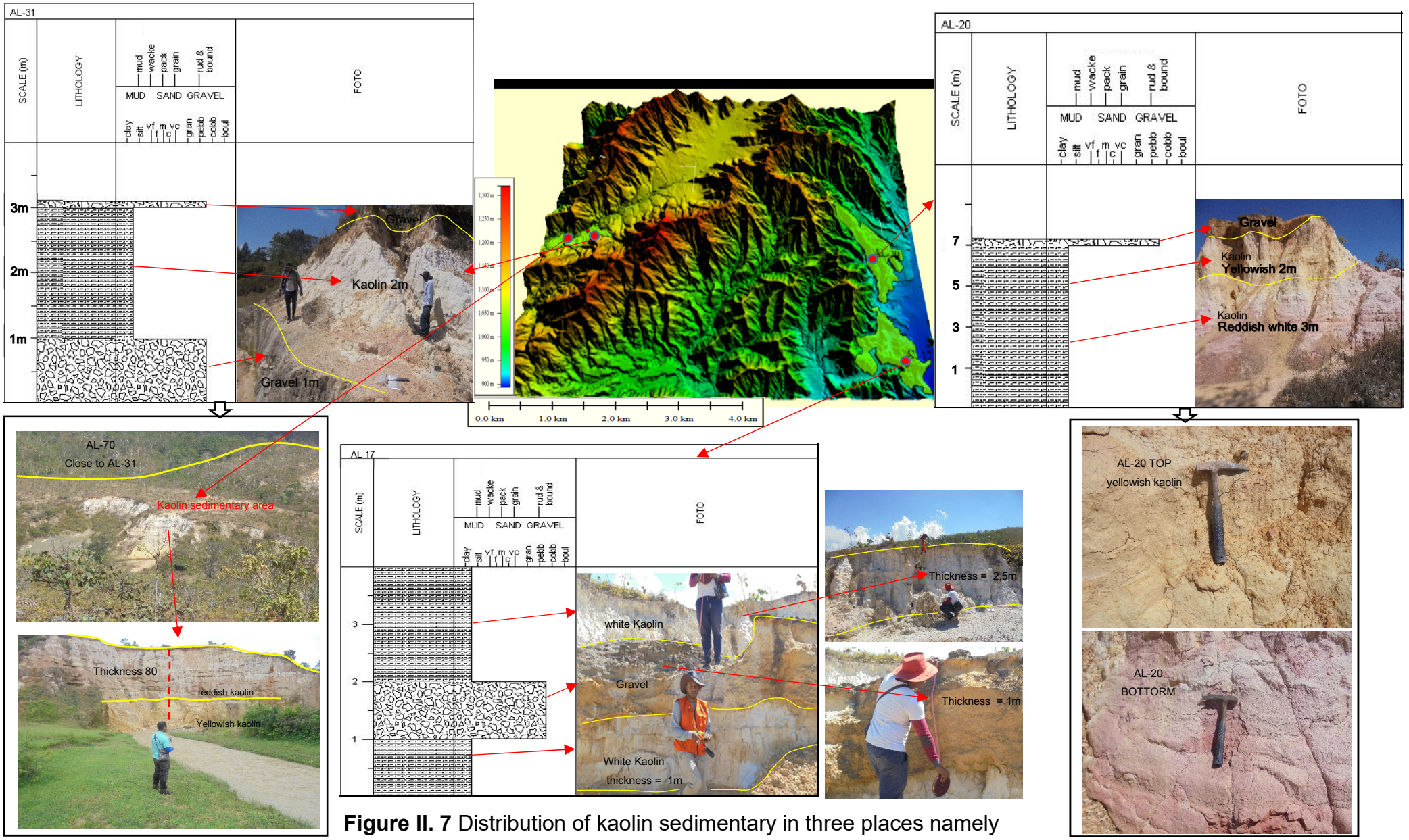

Figure II. 7 Distribution of kaolin sedimentary in three places namely

1. Located at Satu area - in the western part (AL-31 and AL-70)
2. Two other kaolin places located at Kabasfatin area (east - close to Aileu city) (AL-20, AL-17 represented) (Photo IPG, 2019).

CHAPTER III. DEFINITION OF KAOLIN

III.1 Definition of Kaolin

The name “Kaolin” is derived from the Chinese term “kao-ling” which means “high ridge”, a Chinese village near Jingdezhen, in southeastern China’s Jiangxi Province. The name Kaolin entered English in 1727 from the French version of the word “Kaolin”, as we know it now, where kaolin was first explored in the XVII century (Kužvart, 1977).

It has been difficult to define exactly the term "kaolin" (Alves *et al*, 2014). It has the following definition: "Kaolin is, from the petrographic point of view, a clay rock whose essential mineral is kaolinite". This definition undoubtedly expresses a necessary but not sufficient condition for the distinction between kaolin and clay.

Given the astonishing development that has been observed since the 1950s in the study of clay minerals, the merits of the research media developed in the meantime – X-Ray, Infra-red, electron micrographs, thermal methods and even optical and chemical methods – today it is possible to identify and strictly classify, in many cases, clay minerals. In addition, what is most interesting is the possibility of the being able to explain, on a scientific basis, the technological behavior of this raw material in its most varied industrial uses.

Grim (de Souza Santos, 1975) addressing the problem of the definition of kaolin, merely transcribes the definition given by Ross (1931) which states: “kaolin is understood as a rock mass composed of a poor clay material iron and usually white or almost white and one consisting of hydrated silicate of an alumina of about $2SiO_2$. $Al_2O_3.2H_2O$ other bases, if present, being assumed to impart impurity or observed materials.

The French association of kaolin producers, for customs reasons, found it necessary to define the marketable product kaolin. Thus, they define kaolin as a “white or almost white plastic clay of the highest quality used in the porcelain industry and serving as a filler in the paper industry”.

In England, the term kaolin has been used for raw or reclaimed material, with the term “China-Clay” being reserved only for the kaolinite (washed and purified) material extracted from the primary hydrothermal deposits.

In the countries of the socialist economy “kaolin” is defined as a substance of white color or almost of industrial utility and formed “*in-situ*” by chemical decomposition of some type of rocks, especially rocks containing feldspars (Kužvart, 1977).

III.2 Genesis and Occurrences of Kaolin

There are two kinds of formation of kaolin, namely primary and secondary. Primary kaolin which is produced through weathering and hydrothermal alteration and secondary kaolin is produced through transportation and sedimentation occurs in a lower places, in other words, kaolin sedimentary.

III.2.1 Primary Deposits

Kaolin occurs primarily through weathering and hydrothermal alteration. The weathering process is strongly influenced by endogenous processes such as wind, surface water, subsurface water, which fills in fractures in acidic composition up to intermedia.

A. Weathering

The weathering process will occur close to the surface of the ground or very close to the surface of the soil. The formation of kaolin will be strongly influenced by climate, geomorphology, hydrology, chemistry and thermal energy, tectonic conditions and rock composition. Deposits of secondary residues from kaolin are the result of weathering feldspars in a non-base medium.

Climate is one of the main conditioners of exogenous kaolinization. In fact, the ideal climate occurs in weathering which is responsible for the formation of alterations residual kaolinite deposits and tropical climate. Average annual temperatures vary between 16^{o}C and 18^{o}C and annual rainfall is around 1000 mm/m^2 (Kužvart, 1977). In addition, geomorphological conditions affect the position of the water level and,

therefore, drainage of weathered crust. Favorable geomorphological conditions are responsible for the permanent removal of kaolinization products (K^+, Na^+, Mg^{2+}, and some silica). In fact, the main requirements for kaolinization are as follows.

- Low concentration of K^+ and Na^+
- Lixiviation carried out by a medium of pH between 4 and 5;
- Intense lixiviation of Ca^{2+}, Mg^{2+}, K^+, Na^+, and Fe^{2+} (usually formed by the reduction of Fe^{3+} through organic matter;
- Humus resulting from the vegetation composition;
- Presence of H^+ (pH 4-5) and CO_2

The following is the reaction of kaolinite formation due to weathering of potassium feldspar and also due to the reaction that occurs between orthoclasts and carbonates:

$K_2O\ Al_2O_3\ 6SiO_2 + CO_2 + H_2O$	$K_2CO_3 + Al_2O_3\ 2SiO_2\ 2H_2O + 4SiO_2$		
Feldspar	Solution	Kaolin	Silica

B. Hydrothermal Alteration

Hydrothermal alteration is a process that occurs because it is influenced by hydrothermal activity that interacts with a host rock. So after an interaction between the host rock and hydrothermal activity there will be a complex change that involves changes in minerals, chemistry, and texture caused by the interaction of hot liquids with the rocks traversed, under conditions of physiochemical evolution.

Hydrothermal fluid is a hot liquid in the form of magmatic water, meteoric water, connate water, or minerals containing water originating from the Earth's crust produced during the process of metamorphism. The thermal fluid then moves upward with the components that form ore minerals so that mineral and mineral changes occur. Composition alterations occur in rocks when rocks interact with hydrothermal solutions.

From hydrothermal activity below the surface and moving up to the surface and interacting with the source rock there is a replacement process and cavity filling process, then the process of hydrothermal change is characterized by the effect of

hydrothermal solution which can cause changes in mineralogy and texture of stone walls.

The processes that occur due to many hydrothermal changes that play a role in the process of forming kaolin (kaolinization, for example the reaction is as follows:

$K_2O\ Al_2O_3\ 6SiO_2 + CO_2 + H_2O$	$K_2CO_3 + Al_2O_3\ 2SiO_2\ 2H_2O + 4SiO_2$		
Feldspar	Solution	Kaolin	Silica

In the reaction of kaolin formation from feldspar due to the influence of hydrothermal solution such as the reaction above it can be seen that the H_2O component enters feldspar and K_2O (+ some SiO_2) exits. Transfer of elements from hydrothermal solutions at certain temperatures and pressures will cause changes in mineralogy and wall rock texture.

Factors that influence the hydrothermal intensity and alteration results include characteristics and composition of the original rock (host rock), composition of the hydrothermal solution, conditions of temperature, pressure and phase changes of hydrothermal solutions and changes in certain initial elements (such as the release of H_2S into strong acids).

III.3.3 Secondary Deposits

Kaolin sedimentary is a process of continuation of the primary sedimentation process produced through weathering and hydrothermal alteration. So kaolin that has been produced through weathering and hydrothermal alteration processes is then transported by water, wind, ice, or glacier media to a suitable place in the basin, usually these final deposits can be carried out in low-speed rivers, lacustrine, lagoon or delta environments.

From the expert's view of sedimentary kaolin deposition is as follows: according to (Pettijohn *et al*, 2012) secondary deposits or kaolin sediments are produced by the clay sedimentation cycle which is different from other sedimentary cycles because clay (kaolin) is transported in suspension, and it is not completed, and also because its main deposition is by mechanical sedimentation (no chemical or organic precipitation).

The different geological origins of kaolin produce important differences in mineralogical composition (de Souza Santos, 1975). Usually, secondary kaolin gives lower quartz and mica content, but it presents higher contamination of iron and titanium oxide, which is responsible for the original white discoloration (Bristow, 1987a). One of the main characteristics of secondary kaolin is finer grain size of clay minerals. Secondary deposits are common in Georgia (US); Rio Jari (AP) and Rio Capim (PA) (northern Brazil); West Germany; and Guadalajara (Spain).

Secondary kaolin is classified into three types: (i) sediment, (ii) kaolinite sand and plastic clay, (iii) flame retardant and silica. Kaolin sediments, which have a high percentage of kaolinite (above 60%), usually after processing, produce products with specifications suitable for the paper industry (Bloodworth *et al*., 1993). Kaolinite sand contains less than 20% kaolinite. Sand that is rejected in the beneficiation process, in general, is intended for civil development.

III. 3 Physical Characteristic of Kaolin

In general, kaolin is white or rather whitish, hardness 2-2.5, plastic if mixed with water, with electrical conductivity and low heat and specific gravity between 2.60-2.63. The properties of kaolin will be very influenced by the composition of clay minerals present in kaolin, then to find out other physical properties, such as plasticity, strength, texture and others, the properties of the constituent minerals, namely clay minerals, are discussed. According to (Kirsch & Fuchs, 1968) these physical properties include:

1. Flocculation and deflocculation
    a. Flocculation is the process of clumping clay beads into larger lumps, while deflocculation is a process of dispersion where larger lumps become smaller parts.
    b. Flocculation and deflocculation describe the state of aggregation from clay items when mixed with water, where clay minerals quickly absorb water and for kaolin the absorbed water will evaporate on heating at a temperature of 100^{o}C-200^{o}C. The dispersion process can be strengthened by the addition of electrolytes or deflocculants such as water-glass, Na_2CO_3, Na_2PO_4 and etc. The number of uses for

deflocculating a dispersion process depends on several factors (Grim, 1968) among them are the degree of fine grains that show the properties of colloids, the amount and type of dissolved salts present in clays, silicates and electrolytes or deflocculants used, properties of clay minerals.

2. Plasticity

Plasticity is a property that allows clay to be shaped without cracks and the shape will remain after the forming force is lost or removed. The clay will become plastic a few moments later after the clay is mixed with a liquid that has a polar arrangement like water. The clay will not become plastic when interacting with liquids which are not polar like CCl_4. According to Grim (1968), factors that influence the degree the plasticity of the clay include the influence of water, solid materials and the symptoms of colloids that affect the size of solid particles and force pull between molecules, the presence of other materials that affect the particles' properties, orientation of particles in mass, previous history experienced by the materials. According to Grim (1968) kaolinite has a plasticity limit of 25-36.3 which is much smaller than montmorillonite with a plasticity of 86.

3. Thixotropy

Thixotropy or suspension power is a characteristic of clay minerals which when mixed with a liquid will form a suspension. This characteristic is related to plasticity. Fine grained of kaolin will remain suspended in water for hours without showing signs of settling. When flocculants are added, such as acid, borax, $MgSO_4$ etc., there is clumping or flocculation with precipitation which takes place quickly. If it is added to the electrolyte a solution like water-glass or Na_2CO_3 the dispersion process increases and it is produced a more permanent suspension.

4. Texture

The texture of clay minerals includes the size and shape of mineral particles clay which affects its plasticity, mechanical strength, ease in drying and

characterizing the product after burning and kaolin generally has two types of textures (Grim, 1968), namely the texture of non-plastic minerals which are generally the coarse impurities to fine texture and the texture of very fine minerals.

5. Dry Retraction

   During the drying process there is a discharge of water which allows the clay to stick with one another; this is termed as dry retraction, which still has some residual water called pore water that can last up to 110 ° C warming. The clay varies much during dry retraction. The degree of variation in dry retraction clay is identical to the variation in the amount of water needed to cause its elasticity, the higher the density of the clay the more water is absorbed then greater is the dry retraction. Clay which has high value of dry retraction is difficult to dry without cracking; the reduction of the occurrence of cracks or ruptures can be done by the addition of non-plastic materials such as quartz sand, flint and feldspar.

   According to (Gooseff *et al.*, 2002) kaolin dry retraction is divided into 3 categories, namely rough kaolin in water line dry retraction 5.0-7.6, for washed kaolin ranging from 3.3 10.8, and for kaolin sediments ranging from 4.5 to 12.8.

6. Strength

   Factors that influence the dry strength of clay minerals (Grim, 1968), among others, are the size and shape of the grain of a plastic part and non-plastic, the degree of clay flocculation before being burned, the number of smooth items, the length of time and temperature at the time the clay was being pressed (aging) before being formed, the amount of water used to evaporate the mass plastic, a mixture of water and other materials, the method used in evaporates ready-to-use mass, speed and high time temperature drying.

7. Slaking

   Slaking is the nature of clay when it is hit by water and then floats and then it breaks into smaller parts.

8. Color

   The color of kaolin will be influenced by the color of the constituent clay minerals, where the color of clay minerals will be determined by the iron compounds or carbon materials, sometimes also minerals like manganese and titan minerals in sufficient quantities can affect the color of the clay. The color of white or rather whitish kaolin is caused by the constituent clay minerals free from the above impurities. Color from clay minerals before and after combustion sometimes experience changes. For kaolin before and after combustion generally it will remain with the same white, but it can also change slightly to yellowish white.

III. 4 Group of Kaolin Mineral

The minerals included in the kaolin group are kaolinite, dickite, nacrite, and halloysite (Al_2 (OH) $4SiO_5.2H_2O$), which have a greater water content and generally form separate deposits such as the group kaolin minerals:

1. Kaolinite group of common clay minerals that are hydrous aluminum silicates; they comprise the principal ingredients of kaolin (china clay). The group includes kaolinite and its rarer forms, dickite and nacrite, halloysite, and allophane, which are chemically similar to kaolinite but amorphous.

   Kaolinite, nacrite, and dickite occur as minute, sometimes elongated, hexagonal plates in compact or granular masses and in mica like piles. They are natural alteration products of feldspars, feldspathoids, and other silicates. Anauxite, which was previously regarded as a kaolinite-group mineral possessing a higher than usual silica-alumina ratio, is now considered to be kaolinite and free silica (mainly non-crystalline). For chemical formula and detailed physical properties.

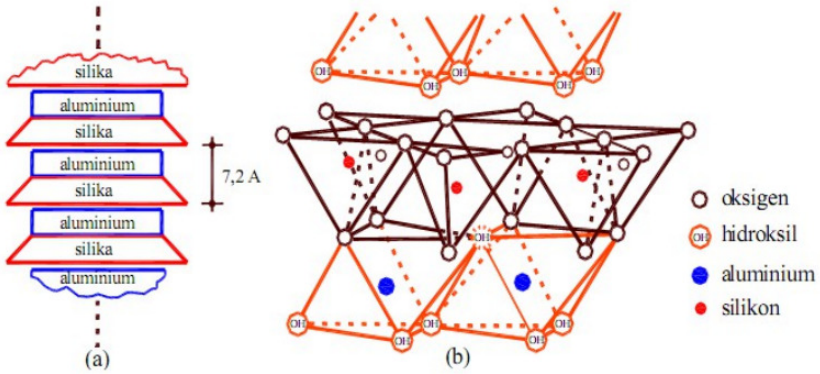

Figure III. 1 Chemical formula and detailed physical properties

2. Halloysite is clay mineral that occurs in two forms: one is similar in composition to kaolinite, and the other is hydrated. Both forms have a lower specific gravity than kaolinite, are fibrous rather than platy, and may exhibit a prismatic tubular shape. Halloysite that has a composition close to that of kaolinite and is characterized by its tubular nature in contrast to the platy nature of kaolinite particles. Although tubular forms are the most common, other morphological varieties are also known: prismatic, rolled, pseudospherical, and platy forms. The structure of halloysite is believed to be similar to that of kaolinite, but no precise structure has been revealed yet. Halloysite has a hydrated form with a composition of $Al_2Si_2O_5(OH)_4 \cdot 2H_2O$. This hydrated form irreversibly changes to a dehydrated variety at relatively low temperatures (60° C) or upon being exposed to conditions of low relative humidity. The dehydrated form has a basal spacing about the thickness of a kaolinite layer (approximately 7.2 Å), and the hydrated form has a basal spacing of about 10.1 Å. The difference of 2.9 Å is approximately the thickness of a sheet of water one molecule thick. Consequently, the layers of halloysite in the hydrated form are separated by monomolecular water layers that are lost during dehydration.
3. Nacrite is a rarely occurring mineral from the mineral class of “silicates and Germanates ". It crystallizes in the monoclinic crystal system with the chemical composition Al_4 [$(OH)_8$ | Si_4 O_{10}], is thus seen crystal chemically an aluminum - phyllosilicate with hydroxide ions ($(OH)_2$) as an additional anions . Nacrite develops mostly earthy, flaky or massive aggregates, rarely also small, tabular, irregularly pseudohexagonal crystals of white, gray or yellowish

brown color and pearly gloss. Nacrite forms in cavities of hydrothermal deposits. Accompanying minerals include calcite, dolomite, fluorite, quartz and topaz.

III. 5 Kaolin Processing and utilization

The processing of kaolin is mainly intended to dispose of contaminant minerals, such as quartz sand, iron oxide, mica and others. Besides that it aims to get fine grains, high brightness (brightness), certain water content, certain pH and other properties. The treatment process is very dependent on the amount of impurity minerals and usage specifications (Sukandarrumidi, 1999). Kaolin is widely used in various industrial fields, both as the main raw material and as an additional raw material. In this case the utilization of the physical properties possessed by kaolin, such as: fineness, strength, color, electrical conductivity is very necessary. As an industrial material, kaolin is widely used in the paper, ceramic, paint, rubber, pharmaceutical, metal and other industries.

CHAPTER IV. METHODOLOGY

IV.1 WORKFLOW

Work plans implemented, both in the field and in the laboratory, based on pre-determined objectives, aim to study and characterize clayey raw materials. This study method can go through several stages such as field work to obtain representative samples, laboratorial preparation, analysis and data interpretation. Bellow is shown the flow chart (figure IV.1) and a more detailed explanation of each phase and methods.

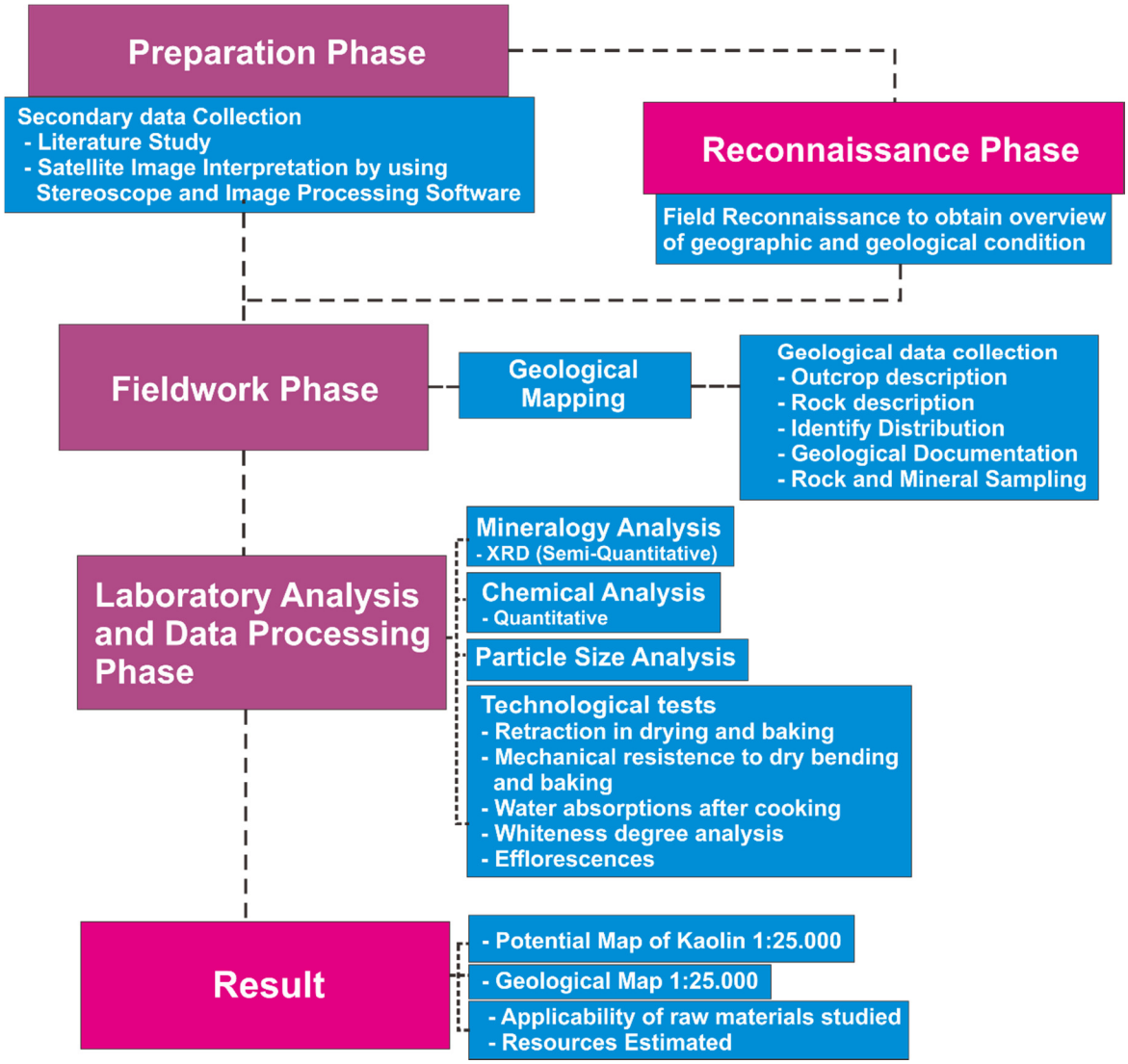

Figure IV. 1 Flow chart of the phase and methods used in this study.

1. Preparation phase

Preliminary studies are carried out with the aim of providing a general description of the geological and geographic conditions of the area, as well as facilitating the sequence of the study activities. Preliminary studies can be divided into several stages, among others:

a. Literature study, before conducting research activities in the field. First it must be obtained and studied data from various sources, such as geological data and mineral excavation material both regionally and locally.
b. Interpretation of satellite imagery using stereoscope and image processing software and topographic maps are important elements in interpreting. The expected interpretation results are in the form of geomorphology, lithology, geological structure, flow patterns and geographical conditions. The results of this interpretation will be reference material for track planning. The trajectory is focused on the distribution of the primary and sedimentary kaolin areas carried out by traversing rivers, cliffs on the roadside and across the hills to obtain more detailed data.
c. Recognize places, directly make observations at the study location with the aim to determine the state of road accesses, general geological conditions and geographical position.

2. Fieldwork phase

The fieldwork activities are carried out based on the methods and concepts that have been planned. To obtain data, it is carried out following the track planning, so that the study area can be delineated as much as possible. Field work consists of geological mapping, mapping of kaolin distribution and potential of excavated materials.

- In geological mapping, the distance between each observation site is about 250 meters. However, outcrop conditions in the field required that, in some places, the distance was more than or less than 250 meters. Field elements include lithological data, clay minerals, geological formations and lithological contacts, stratigraphic measurements and morphological observations.

- Data obtained from the field in mineral mapping activities, including rock sampling and clay mineral samples (kaolin).

3. Laboratory analysis phase

The laboratory work took place at the CTCV (Coimbra, Portugal).

The CTCV (“Centro Tecnológico da Cerâmica e do Vidro”) (Figure IV.2) is an **Entity of the Scientific and Technological Portuguese System**, it is certified by CERTIF – “Associação para a Certificação” according to NP EN ISO 9001 and with laboratories accredited by IPAC (“Instituto Português de Acreditação”) according to the norm NP EN ISO / IEC 17025, for performing analysis and tests. It is also a “**Sectorial Standardization Body”** recognized by the IPQ (“Instituto Português da Qualidade”), with an active participation in the **National, European (CEN) and International (ISO) Standardization Technical Commissions.**

Figure IV. 2 CTCV (Centro Tecnológico da Cerâmica e do Vidro)

It is composed by a multidisciplinary team with solid scientific and technical skills in different areas of knowledge, which are the result of its 30 years of accumulated experience and specialized training. With a R&D culture oriented towards results and value for the industry, it offers its business partners a set of integrated solutions ranging from specialized consulting and auditing, to professional training, to measurement and testing and to RTD solutions. The services of CTCV are oriented towards the Ceramics and Glass and the whole cluster of the habitat.

The CTCV's performance is essentially based on the partnership it develops with clients, other entities of the Scientific and Technological System and Sectorial and Regional Associations, as well as in the rigor and credibility, allied to the strong innovation and technology transfer component.

The Materials Analysis Laboratory (MAL) is an analytical laboratory for the quality control of mineral raw materials, non-metallic inorganic materials and other industrial materials. The diversity of the performed tests is one of the main differentiating factors of MAL, covering a wide range from the characterization of materials to the environmental and industrial hygiene controls. Within the accreditation scope of the laboratory there are 62 accredited tests which cover the following technical areas:

- Aggregates and Inert materials;
- Asbestos
- Ambient Air
- Concrete, cement and mortars
- Gaseous effluents
- Liquid effluents
- Solid waste
- Ceramics and glass

The laboratory guarantees solid technical competence, based on specialized and experienced human resources and offers a wide range of equipment. The CTCV team is qualified for controlling the quality of several matrices, according to the analytical methodologies described in national or international standards, or even through internally developed methodologies, duly validated.
Regarding Quality Control, MAL's goal is the reliability of obtained results and actions of Internal and External Quality Control are developed, using the regular use of Certified Reference Materials and the continuous participation in various programs of international inter-laboratory testing.

The tests and analyses that MAL carries out are, among others:

- Quantitative chemical analysis of aggregates and concretes (EN.1744-1).
- Determination of pH, Conductivity, Total Suspended Solids (SST), Chemical Oxygen Demand (CQO), Biochemical Oxygen Demand (CBO_5), Fluorides, Sulphates, Chlorides in liquid effluents.
- Chemical characterization of waste, for landfill purpose (Dec. Law nº 183/2009).
- Evaluation of air pollutants in suspension in ambient air (dust, crystalline silica, asbestos fibers).

- Determination of the heavy metal content, Particles, SO_2, fluorides, chlorides and H_2S in gaseous effluents.
- Determination of the chemical composition of raw materials (sands, carbonates, clays, feldspars, calcites, kaolin's, dolomites, chromites, plasters, etc).
- Determination of Pb, Cd, Cr (VI) and Hg in packages and package waste glass, covered by the directive 94/62/CE
- Determination of the solubility lead and cadmium in products containing food (EN 1388-1, ASTM C 738, ISO 6486-1, ISO 7086-1)
- Tests for the certification of crystal glass and sonorous.
- Granulometric analysis of a wide range of powder materials, including natural and synthetic ceramic powders, pigments, metal powders and others
- Determination of real density of powders and compacts.
- Differential thermal analysis and thermo gravimetric.
- Dilatometric analysis.
- Technological tests: retraction determination, flexural strength and water absorption after drying and firing, of samples raw materials or ceramics composition.
- Identification of crystalline phases by X-ray diffraction
- Characterization of thermal behavior of materials by heating microscopy (up to 1700ºC).

The main technical means of the laboratory include:

- Scanning electron microscope with EDS system
- X-Ray diffractometer
- Heating microscope
- Thermo balance
- Dilatometer
- Calorimeter
- Microbalance
- Sedimentation and X-ray granulometry analyser
- X-ray fluorescence spectrometer
- UV/VIS spectrophotometer

- Atomic absorption spectrophotometer
- ICP

The analytical program that was performed at CTCV on kaolinitic samples was as follows:

- Mineralogy analysis
- Chemical analysis
- Particle size analysis
- Determination of the level of in raw and firing
- Rheology test (deflocculation curve, wall formation rate)
- Test drying and roasting technology at three temperatures, with determination:
  - ✓ Retraction when drying and firing
  - ✓ Mechanical resistance to dry arches and combustion
  - ✓ Absorption of water after firing
  - ✓ Evaluate colors, textures and defects

Information on analytical methods such as mineralogical analysis, chemical analysis, particle size analysis and technological tests mentioned above will be explained bellow.

4. Result

Result is the final activity that summarizes all the results and interpretation of data from the initial to the final stages such as collecting secondary data, mapping in the field, sampling, laboratory analysis, interpretation of data and the final writing of the dissertation. The final report provides the geological information of the study area and studies of the characteristics of the kaolin and provided resource estimation of the special kaolin clays and their industrial use.

IV.2 METHODS

The purpose of this analysis is to determine the characteristics and quality of the samples. The techniques and parameters used for this analysis are as follows:

1. Mineralogical analysis

Minerals are the constituent materials of rocks, so every presence of minerals in rocks determines the naming of rocks and the characteristics of a rock. So the purpose of this mineral analysis is to find out what constituent materials and minerals are contained in these rocks. Kaolin belongs to a group of clay minerals consisting of both clay and non-clay minerals as an indication of impurities such as (quartz, calcite, pyrite and feldspar) and organic and salt solutions. So mineral analysis is one of the requirements to find out the quality of kaolin for the industrial needs of ceramics, paints, pharmaceuticals, rubber, and other ornamental items. XRD (X-Ray Diffraction) is one of the most effective analysis methods used in describing rocks and certain chemical compounds in solid form using diffraction/X-ray reflection. X-rays are electromagnetic waves produced by sudden high-speed particle decelerations (Moore and Reynold, 1997). Mineralogical analysis by X-Ray Diffraction is an analytical method that is effective in describing rocks and certain chemical compounds in solid form because the preparation process is easy, cheap and fast.

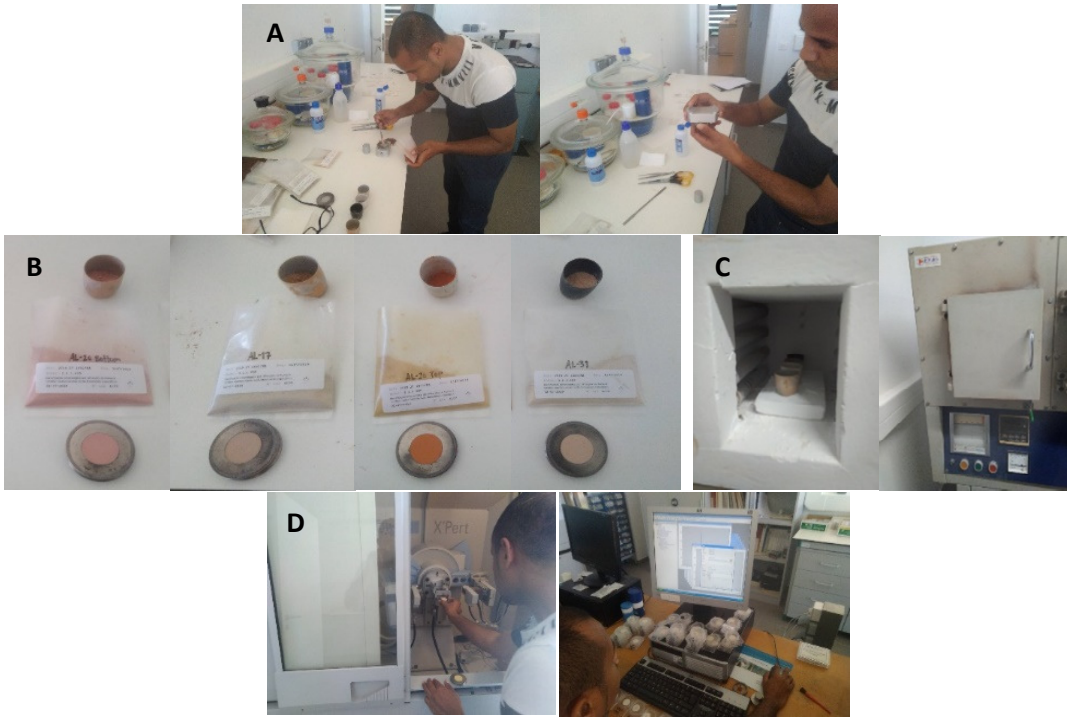

Figure IV. 3 (A) Sample preparation to put the sample into tube.
(B) Dried samples ready to be analyzed.
(C) Drying process with a temperature of 40°C for 35 minutes
(D) Equipment for mineralogical analysis “PANalytical X-ray diffractometer, the X'PERT-PRO model” for analysis.

From the first stage of sample preparation to the analysis, the sample was dried in a drying chamber at 40 ºC for 35 minutes. After that the sample was sieved to get a portion below 75 microns (200mesh), then weighed to get enough quantity for this mineralogical analysis (about 30 g). Of these 30 g, smaller sub-samples weighing 5 g were taken. In order to make a comparison of the results, two 5 g samples are made for mineralogical analysis. After that each 5 g sample is put into a mineralogical detection device such as the PANalytical X-ray diffractometer - X'PERT-PRO model for analysis (Figure IV.3).

2. Chemical analysis

The chemical analysis method used is a semi-quantitative analysis which aims to find out the amount of an element or compound in a sample to be analyzed. Analysis of kaolin samples will be done quantitatively and XRF is a tool used to analyze the chemical composition and concentration of the elements contained in the sample by using spectrometry. The purpose of analyzing chemistry in kaolin samples is to determine the chemical composition in order to determine the characteristics of kaolin and also to determine the quality of kaolin. The test includes chemical analysis of the sample and this test is carried out to determine the quality of the kaolin minerals, especially the chemical composition of the 4 samples of different colors. The results of the chemical analysis of the sample will be compared with the requirements for the standards that are used as a reference for existing kaolin raw material specifications.

Figure IV. 4 (A) Samples sieved at 200 mesh ASTM (75µm)
(B) The sample to be dried at 1000 ± 100 ˚C for 1 hour
(C) Put and allow to cool in a desiccator to room temperature
(D) Spectroflux 110, lithium tetraborate
(E) Weigh 10 grams of spectroflux 110, lithium tetraborate
(F) Dried Spectroflux 110, lithium tetraborate at 100-110 ˚C
(G) Mixture kaolin sample with lithium tertaborate
(H) Add to the mixture about 1 ml of lithium bromide in 5% solution
(I) Equipment Call Perl'X 3 (IT 311.017) to prepare samples like pearl cup for chemical analysis.

The initial stage of this process begins with drying in a dryer at a temperature of about 110°C, after which the sample size is reduced by a milling machine (Moínho de Anéis), then sieved to get a standard 200 mesh ASTM (75 µm) size, then weigh 30 grams for chemical analysis (Figure IV.4).

The second stage of preparation of vitreous specimens for chemical analysis:

1. Take a few grams of kaolin sample and calcinate with a temperature of 110 ˚C for 1 hour after it is removed weighing 1 gram of kaolin sample and then calcinate again with temperature and one hour after weigh again to confirm that the difference in weight does not exceed 4% .

2. Weigh 10 grams of spectroflux 110, lithium tetraborate, and then mix with 1 gram of calcined kaolin sample.
3. Add to the mixture about 1 ml of lithium bromide in a 5% solution. Close the bottle tightly and shake well to ensure homogenization of the mixture.
4. Pour the mixture into platinum wax (Call Perl'X3 (IT 311,017)) Place the container in a refractory cup and the cup on a hook. When the pearl cup cools, it is removed from Perl'X3 and not printed; the pearl is placed in a parchment envelope by noting only the analysis number. The sample is ready for analysis.

3. Granulometric analysis

The granulometric analysis is important in the characterization of ceramic materials, since the particle size and its granulometric distribution influence its technological properties and consequently are determinant factors in the application of kaolin. In a sample, the particles do not all have the same size and therefore it is necessary to obtain information concerning the dimensional distribution of these particles and thus to realize the calibration of the sample and determine the dominant dimensional classes. The shape of the particles can influence the distribution, in that elongated particles can be accounted for in the finer fractions because they have smaller cross-sections than the longitudinal sections, i.e. they can pass through sieves with small meshes. Therefore, when referring to the size of a particle, the value is always relative to an ideal spherical dimension, called the equivalent spherical diameter (e.s.d.). The Wentworth scale enables the samples to be classified according to the predominant s.d. Table 3 lists the s.d. ranges for each dimensional class.

Table IV. 1 Classification of the particles taking into account e,s,d. according to the Wentworth classification, with emphasis on particles of e.s.d. <63 μm. (adapted from McLane, 1995).

Equivalent spherical diameter	**Classification**	
2 ≤ cm <25,6 0,0063 ≤ cm ≤ 2	Phenoclasts Sand	
31 ≤ μm <63 15,6 ≤ μm < 31 7,8 < μm ≤ 15,6 2 < μm ≤ 7,8	Silt	Coarse Silt Medium silt Fine silt Very thin silt
μm < 2		Clay

Figure IV. 5 (A) Preparation of 500g for each sample to granulometric analysis
(B and C) Preparation and drying in a dryer at 110°C for 24 hours
(D) Weighing 50 g of 500 g for each sample
(E) Mixed with 200 ml hexametaphosphate liquid and then shaken and leave it for 24 hours
(F) Determination of residuals; Fraction<63µm and fraction>63 µm separated on a wet sieve
(G) Dried residuals material (fraction<63 µm and fraction>63 µm) in a dryer at 110°C for 24 hours
(H) Two samples (fraction<63 µm and fraction>63 µm) prepared for granulometric analysis.

The initial stage of sample preparation for grain size analysis is 500 g of material. Then the kaolin sample is dried in a dryer at 110°C for 24 hours. 50 g of the 500 g are weighed and mixed with 200 ml hexametaphosphate liquid and then shaken and stirred until completely mixed, after they are left for 24 hours to settle. After 24 hours the sample mixed with water will be made to pass a set of sieves in the range of 5600 µm down to 63 µm (Figure IV.5). If the percentage of fraction residuals (sand) is more than 2 % then the analysis will be carried out by sieving, if the percentage of fraction residual is less than 2 % then fraction analysis will be carried out by sedimentation (SediGraph III equipment) to obtain a complete distribution.

After preparation with sieving, the samples are separated by fractions > 63 μm and fractions <63 μm, then dried in a dryer at 110°C for 24 hours. After drying the sample, the separation of the fraction <63 μm is carried out in a wet sieve using a 63 μm mesh screen ASTM E11. After analyzing (SediGraph II) the fraction <63 μm, fractions <45 μm and <2 μm were estimated from the absolute frequency of each grain size distribution, turned into an integral sample percentage. This procedure was carried out during the preliminary (recognition) and definitive tests (2 samples were selected), because it was necessary to repeat the procedure to adjust the blurring limits used in granulometry.

Residual samples with a fraction of >63 μm are weighed to get a total percentage of the 50 g sample. The treatment as described has also been carried out on duplicate samples to make a comparison, the difference between the two samples should not be greater than 4%.

4. Technological Test

Beside chemical and mineralogical analysis and particle size analysis on kaolin, analysis of physical properties testing through technology testing is also necessary to determine the character and quality of kaolin (clay mineral) which will be crucial for the industry needs. For the analysis of the technological properties, the amount of sample needed is 2.5 kg.

These tests are used to analyze the rheological behavior of the raw materials and after firing the raw materials, to evaluate their suitability for the various phases of the industrial processes (raw rheological behavior) and final products (after firing). Dry behavior is analyzed by linear retraction and mechanical flexural strength (MFS) before firing at different temperatures; and firing behavior was also verified by linear extraction, absorption of water and MFS at temperatures of 850 °C, 950 °C and 1050 °C.

To perform retraction, mechanical flexural strength (MFS) and water absorption tests, 10 sample specimens are needed, so that a total of 40 specimens for each sample to be tested, measuring about 10 cm and trapezoidal sections (Figure IV.6). The following is an explanation of the initial stages of the sample preparation process to test the physical and technological properties of kaolin.

Figure IV. 6 (A) Weigh 250g of kaolin sample

(B) Hammer mill as equipment tool to reduce the sample size

(C) Shaker tool for stirring water (2 liters) with kaolin (250g)

(D) Drainage of water on the kaolin sample with gypsum tube

(E and F) kaolin in a moist state in the gypsum tube and ready to be formed into specimens

(G) "Planetary mill" tool is used to make specimen samples for technological tests

(H, I and J) Specimen sample results; 40 specimens are required for each sample.

4.1 Retraction (shrinkage)

Retraction is the contraction experienced by the ceramic piece after water loss by drying out or by modifying the minerals involved after firing along a long axis. That is, it is the relationship between initial and final body length after drying and or after firing. This test aims to analyze variations in the size of the ceramic body either after drying or as a finished product (fired), so that the size of the mold can be optimized to get the final product in the desired dimensions. The following is an explanation of the process of the initial stages of sample preparation through retraction testing (Figure IV.7).

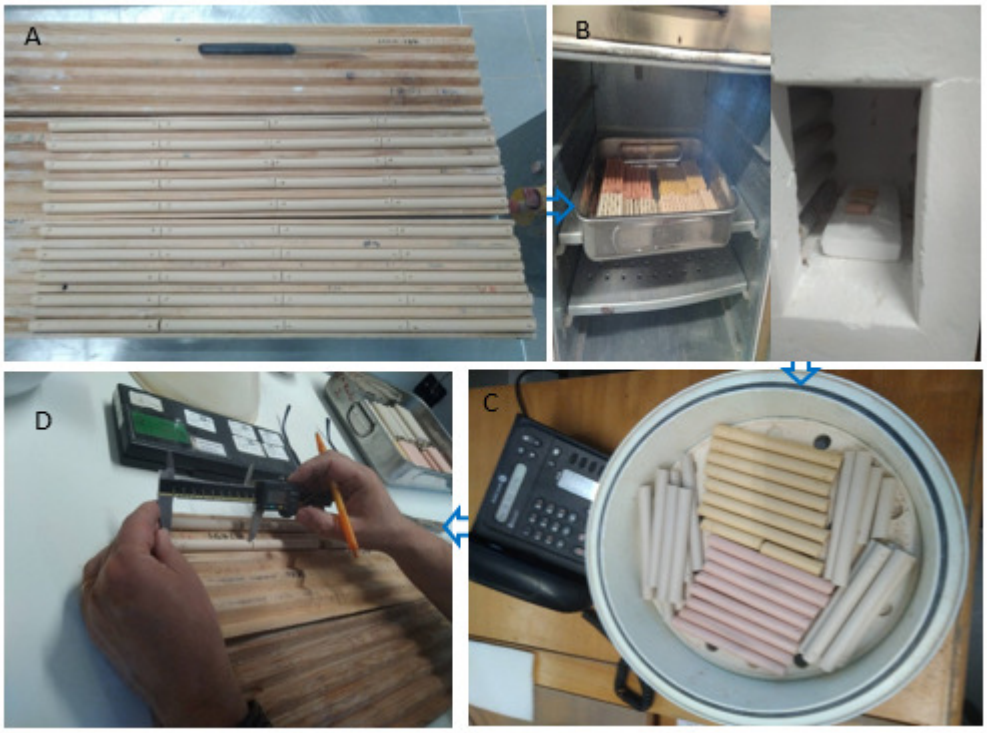

Figure IV. 7 (A) Initial specimens with a size of 10 cm

(B) The specimens are dried in a dryer at 110°C and fired at 850°C, 950°C, and 1050°C. C. Cooling tube to cool the specimen samples to be tested

(D) Measuring the length of the specimens after dried and firing at different temperatures.

The early stages of the preparation of these samples start with the preparation of the specimens, creating as many as 8 specimens with a length of 10 cm. Then they are dried and fired at different temperature: drying at 110 °C and firing at temperatures of 850 °C, 950 °C and 1050 °C. After that the sample specimens are placed in the cooling tube for 20 minutes and, after cooling, they are measured to determine the changes in the samples.

4.2 Mechanical flexural strength (MFS) in dry and after firing

Mechanical flexural strength (MFS) is the load per unit area that the ceramic body (specimen) withstands before breaking and its result is expressed in kgf/cm^2. This property is evaluated after drying and after firing the test pieces at different temperatures (110 °C, 850 °C, 950 °C and 1050 °C). When dried, the MFS is used to verify the strength of the pieces during handling in the industrial process. When fired,

the MFS is of extreme importance as it characterizes the strength of the final product, so that is one of the requirements for the application.

(Table IV.2) The following are the standards or parameters used to determine clay plasticity and porosity:

Table IV. 2 MFS values from which one can infer about the plasticity and porosity of a clay.

MRF (kgf/cm^2)	Plasticity	Porosity
< 30	Low	Elevated
30 – 70	Normal	Normal
>70	High	Low

Figure IV.8 (A and B) Samples that have been dried (110 °C) and fired at different temperatures (850 °C, 950 °C and 1050 °C); (C, D) Mechanical flexural strength (MFS) test.

The tested sample begins with drying and firing at different temperatures with the aim of knowing the characteristics and quality of the kaolin when it is formed or made into a finished material such as ceramic or other ornamentation. From each different

temperature, the temperature that can produce a ceramic that has good quality and strong durability, because a quality requirement for kaolin in the ceramics industry is that it must be resistant to a pressure, resistant to loads and collisions (Figure IV. 8).

4.3 Water Absorption

Water absorption capacity is an indirect measure of porosity of the ceramic body, because it is determined by the percentage of water that can be absorbed by the body. After undergoing a combustion cycle, ceramic bodies may still have porosity and permeability. The water absorption capacity serves as a measure of porosity and permeability that is present after each cycle, to verify when the mineral modification phase allows filling of cavities, making the final product impermeable. If mineral modification occurs at low temperatures and the voids are filled, then the water absorption capacity will be lower; on the other hand, if the mineral modification only occurs at high temperatures (refractory minerals), then the absorption capacity will be greater. The following is an explanation of the process of the initial stages of sample preparation through testing (Figure IV.9).

Figure IV. 9 (A) sample that has been fired at different temperatures of 110 °C, 850 °C, 950 °C and 1050C; (C, B) Determination of water absorption in firing specimens; (C, D) Weighing each specimen before and after it is fired.

This test was performed according to the internal standard of the CTCV laboratory in Coimbra. After testing the mechanical flexural strength, which causes the specimen to fracture, 3 parts were chosen from each set of fired specimens (850 °C, 950 °C and 1050 °C) and let to dry to constant mass (for 12 hours). + 6h). They were then

dipped in boiling distilled water for 2 hours. When the vessel cooled, the parts of the specimens were removed and weighed. The water absorption value was determined with equation 1.

$$\text{Absorption} = \frac{\text{Msat}-\text{Mdry}}{\text{Mdry}} \text{x}100 \qquad \text{Equation 1}$$

Where :

M_{sat} = is the mass of the sample saturated with water,

M_{dry} = is the constant mass of the specimen (dry)

5.1 Whiteness Degree

One important parameter that really determines the sale value and kaolin application is the whiteness value. The whiteness index of a material is influenced by the levels of impurities present in the chemical and mineral.

The sample for whiteness degree analysis at the beginning of the preparation is the same as the chemical analysis, which is first dried in a dryer with a temperature of 110°C then sieving with a size of 75 microns and then weighed to get 10 grams of sample for analysis purposes.

This test is carried out on powder samples by drying and pieces by firing. Pieces of firing samples at temperatures of 850°C, 950°C and 1050°C, and tests were carried out on powders with a drying of 110C, with the aim of analyzing the whiteness degree of different temperatures.

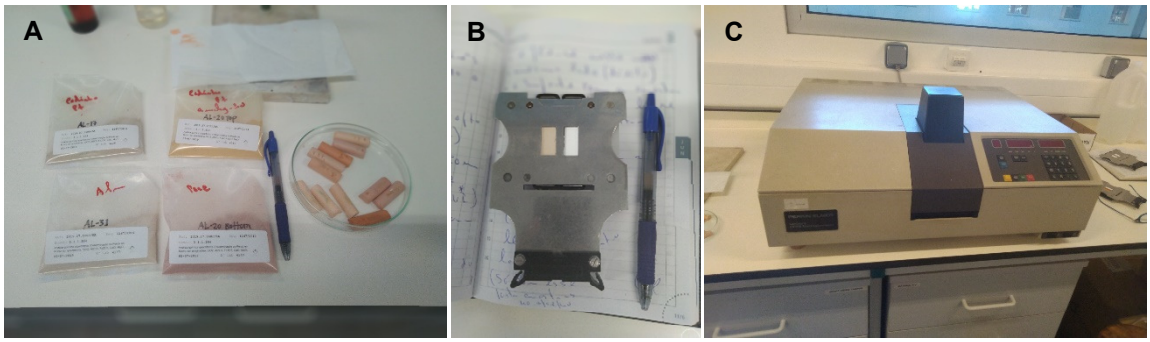

Figure IV.10 (A). Pieces and powder sample after dry and firing ready to analysis.
(B). Ball system using a standard Barium Sulphate plate as a reference.
(C). A UV-VIS spectrophotometer as equipment that use to determine whiteness degree.

6.1 Efflorescences

In chemistry, efflorescence (which means "flowering" in French) is the transfer of salt to the surface of a porous material, where it forms a layer. Important processes include dissolving salts that are stored in water, or sometimes in other solvents. Water, with salt that is now stored in solution, migrates to the surface, then evaporates, leaving a layer of salt.

Testing of efflorescences with temperature (850°C, 950°C, and 1050°C) with the aim to manifest efflorescences when producing a final product. Because the presence of efflorescences on the surface of the ceramic body greatly affects the quality of ceramics.

Efflorescence can clog pores of porous material, causing damage to the material by internal water pressure, as seen in spalling bricks that are firing at different temperatures.

CHAPTER V. RESULTS AND DISCUSSION

This chapter will present the results of laboratory analysis and a discussion of the results of laboratory analysis to determine the characteristics and quality of kaolin samples for application of these raw materials to the ceramic industry.

V.1 Quantitative chemical analysis

The following results are the results of a semi-quantitative chemical analysis of the four kaolin samples by using X-ray fluorescence (XRF). The chemical results show that the composition of the oxide compounds that are classified in clay minerals because it is dominated by Al_2O_3, and SiO_2. The results obtained are shown in the (table V.1).

Table V. 1 Result of chemical composition of kaolin sample

Chemical Composition	4126/19 (AL-17)	4160/19 (AL-20 B)	4143/19 (AL-20 T)	4177/19 (AL-31)
SiO_2 (%)	74,4	69,5	71,8	70,7
Al_2O_3 (%)	17,8	21,4	17,7	21,4
Fe_2O_3 (%)	3,0	3,3	5,9	2,5
CaO (%)	0,0	0,0	0,0	0,0
MgO (%)	0,8	0,8	0,7	0,8
Na_2O (%)	0,3	0,1	0,1	0,1
K_2O (%)	3,5	4,5	3,5	3,7
TiO_2 (%)	0,8	0,9	0,8	0,0
MnO (%)	0,0	0,0	0,0	0,0
P_2O_5 (%)	0,1	0,0	0,1	0,1
LOI (%)	4,7	5,2	5,1	5,9

Samples containing oxides, namely SiO_2, Al_2O_3, Fe_2O_3 and K_2O are consistent with the mineral composition identified (dominant minerals kaolinite and quartz groups). Fe_2O_3 values are generally high, which is more than 1% starting from (2.6% - 3.2%), and the higher value AL-20T is 5.9%. The remaining oxide levels (MnO, MgO, CaO, Na_2O and P_2O_5) are low (<0.9%), where CaO, TiO_2, MnO, and P_2O_5 are less than 0.1%, and MgO and Na_2O range from 0.15% and 0.79%.

V.2 Mineralogical analysis

The mineralogical study was based on X-ray diffraction (XRD) determinations carried out on the powder sample. For semi-quantitative analysis of the powder samples composition, the different minerals were estimated by specific software. The peaks recorded in diffractograms were used for the identification of different minerals, taking into account the characteristic crystallinity and degree of disorder of them.

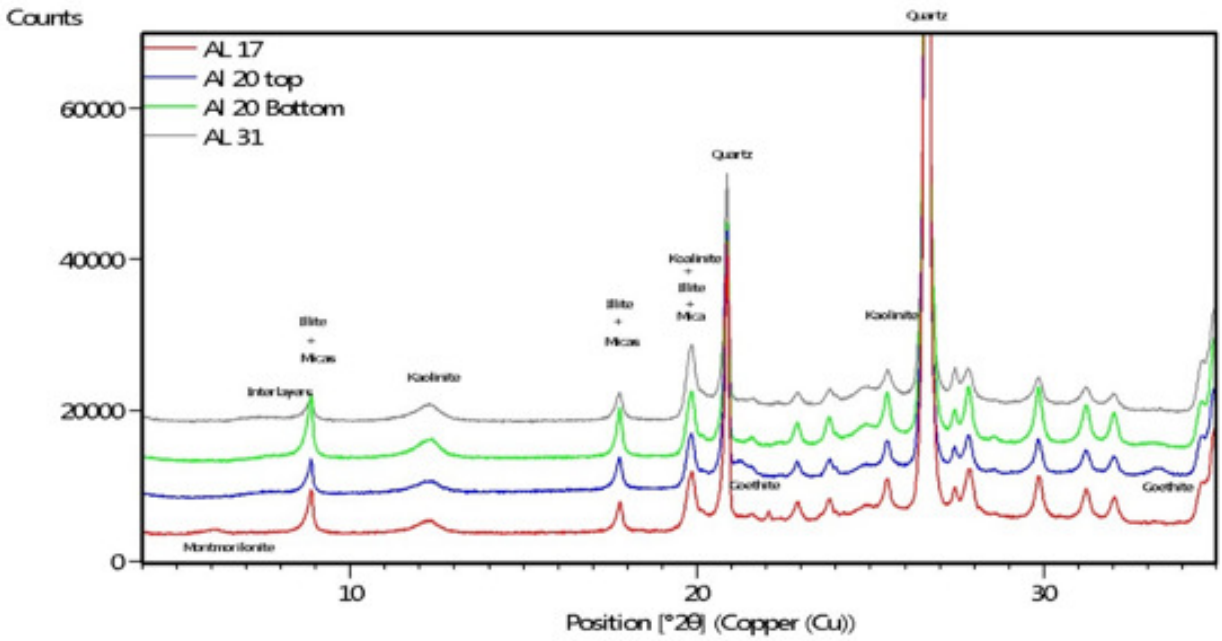

Figure V. 1 Diffractograms of the powder fraction of samples.

When viewed from the graph the highest peak is quartz, followed by illite, kaolinite, montmorillonite (in very small amounts) but there are also other non-clay minerals such as muscovite, and goethite. The highest peak of quartz is caused by low to moderate crystallinity of kaolinite and may be due to the fact that kaolinite crystals may be very thin. The spectra showed the peaks of illite at 2θ = 8.9 and 17.9, kaolinite at 2θ = 13.30 and 25.57. In addition there are impurities fractions such as quartz which are shown at 2θ = 20.85 and 66.50, muscovite at 2θ = 8.9 and 17.9. Goethite as an indication of oxide shows at 2θ = 22.10 and 34.50.

V.3 Grainulometric analysis

The size of the kaolin grains is extremely important not only because it affects other properties as important as viscosity, plasticity, water absorption, mechanical strength and retraction but also because it is part of the requirements for quality.

This analysis was performed on a grains size sedimentation by X-ray absorption (SediGraph III.5120 Mic) and wet sieving. This method is much faster and no less rigorous than standard pipette sedimentation processes, or using a sedimentograph (D.Robbe and R. Bertrand, 1978). The following are the results of the analysis through sedimentation and wet sieving (Figure V.2).

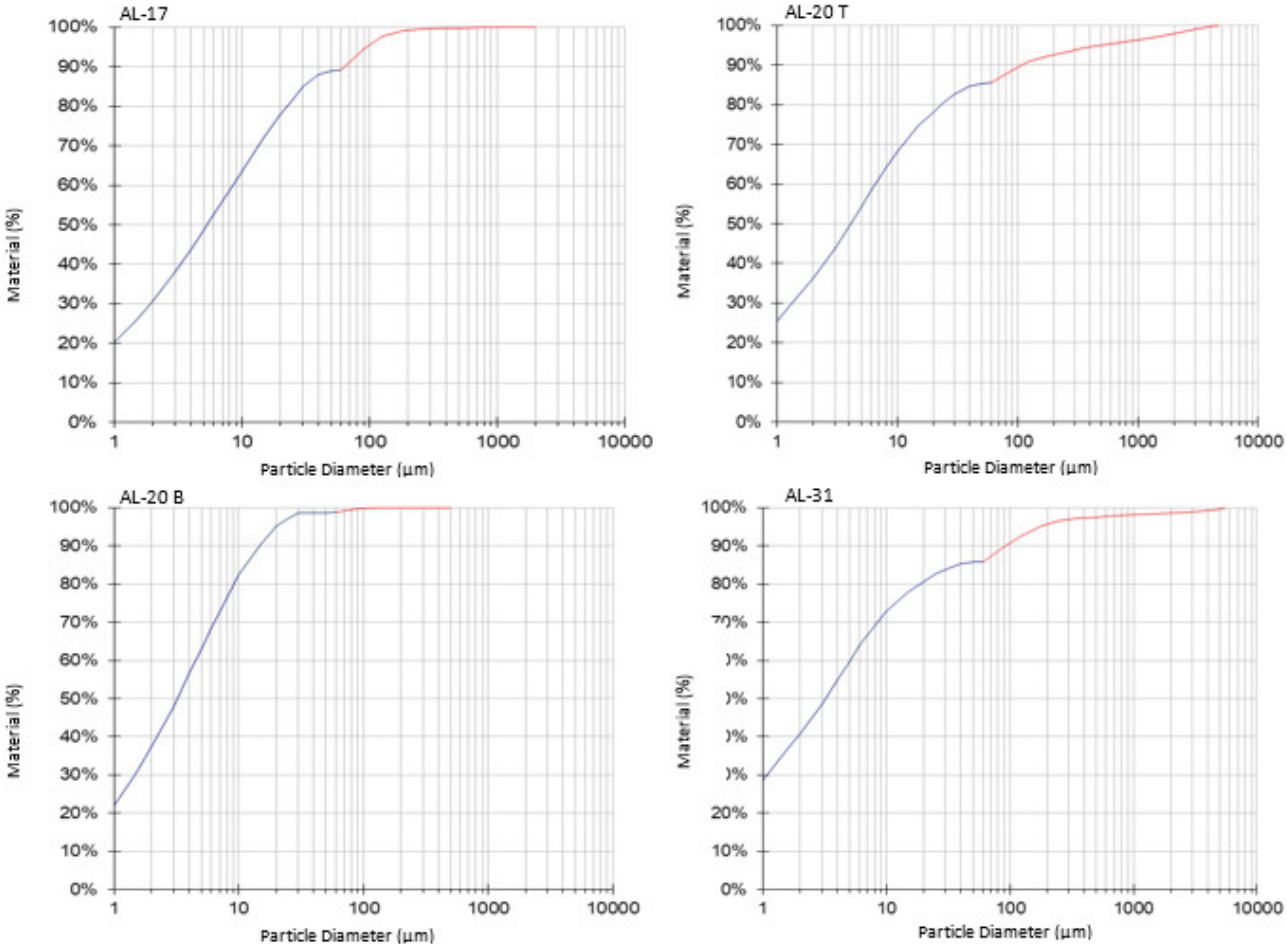

Figure V. 2 Graphic show curve on a grains size sedimentation (blue) and residual particle size wet sieving (red) result.

The grain size distribution was determined for fractions > 63 µm (sand) using wet sieving, in accordance with ASTM C371 standard, and the fraction<63 µm (silt between 2 – 63 µm and clay < 2µm) was analyzed using SediGraph III 5120 micrometrics (IT 3111.102). Below result of grain size distribution (Table V.2).

Table V. 2 Grain size distribution of kaolin deposits

Grain size (%)	AL-17	AL-20B	AL-20T	AL-31
Sand (> 63µm)	10.7%	1,1%	14,4%	14,1%
Silt (2 - 63µm)	58,7%	61,5%	49,4%	45,1%
Clay (< 2µm)	30,6%	37,4%	36,2%	48,8%
D50 (µm)	5.35	3,23	4,12	3,23

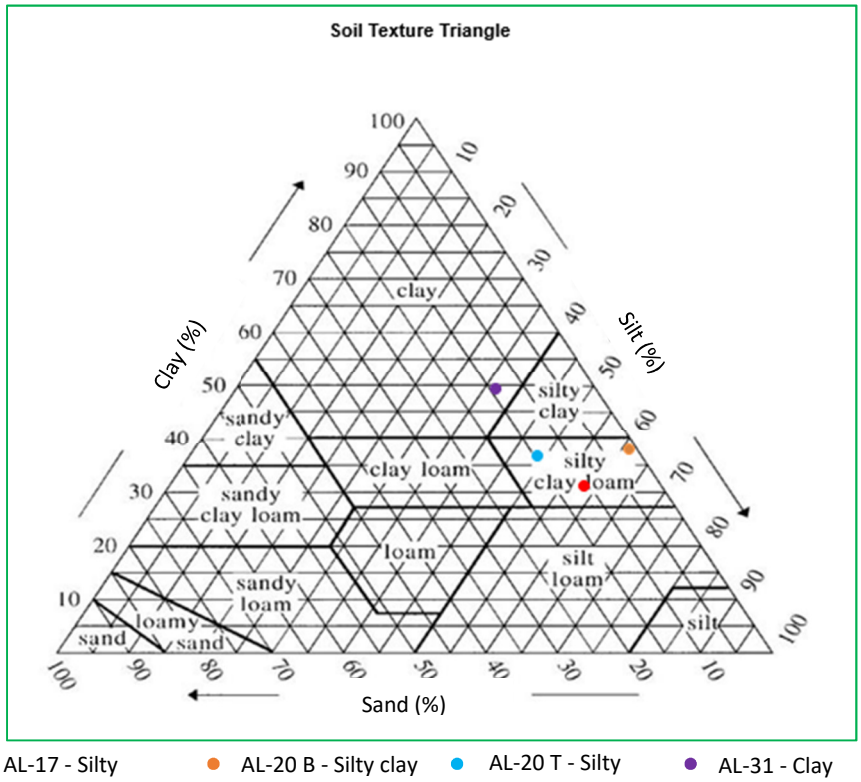

Figure V. 3 Grain size classification diagram based on Natural Resources Conservation Service (NRCS).

The results of grain size classification diagram above based on Natural Resources Conservation Service (NRCS) (Figure V. 3) indicates that the sample of kaolin grain size qualifies as a silt-clay, except AL-31 which belongs to the grain size of clays.

V.4. Technological Tests

From the results of technology testing such as retraction testing, mechanical resistance and flexural and water absorption with samples dried at a temperature of 110° C, and after firing at temperatures of 850°C, 950°C and 1050°C, and on the kaolin samples, shows that these three properties are interrelated with each other,

because the higher the ceramic body temperature, the greater the total retraction and MFS and the lower the water absorption (Table V.3).

Table V. 3 Result of Retraction, flexural strength (MFS) and water absorption tested with difference temperatures.

	Retraction (%)				MFS (kgf/cm2)				Water absorption (%)		
Sample	**Dry 110°C**	**850°C**	**950°C**	**1050°C**	**Dry 110°C**	**850°C**	**950°C**	**1050°C**	**850°C**	**950°C**	**1050°C**
AL-17	9,4	9,4	9,4	9,3	25,4	36,9	64,2	123,7	10,3	9,9	10,3
AL-20B	9,4	9,5	9,4	9,2	4,9	12,4	23,4	98,1	8,4	8,3	8,5
AL-20T	9,3	9,4	9,4	9,2	5,4	9,5	20,3	78,2	9,2	8,9	8,9
AL-31	9,3	9,3	9,3	9,1	13,6	29,8	61,8	137,5	8,9	8,9	8,6

V.4.1 Result of Retraction

Sample pieces from drying and firing at different temperatures. The initial length of the formed sample pieces was 10 cm, and after being dried and fired the sample were measured again and the length of the sample pieces checked to determine any change in size or retraction of the sample. Following is the process of measuring each sample piece (Figure V.4).

Figure V. 4 Measuring the retraction at sample pieces after dried and fired

This chart shows the values corresponding to the total retraction, for each sample, dry and firing of the pieces with different temperatures (Figure V.5).

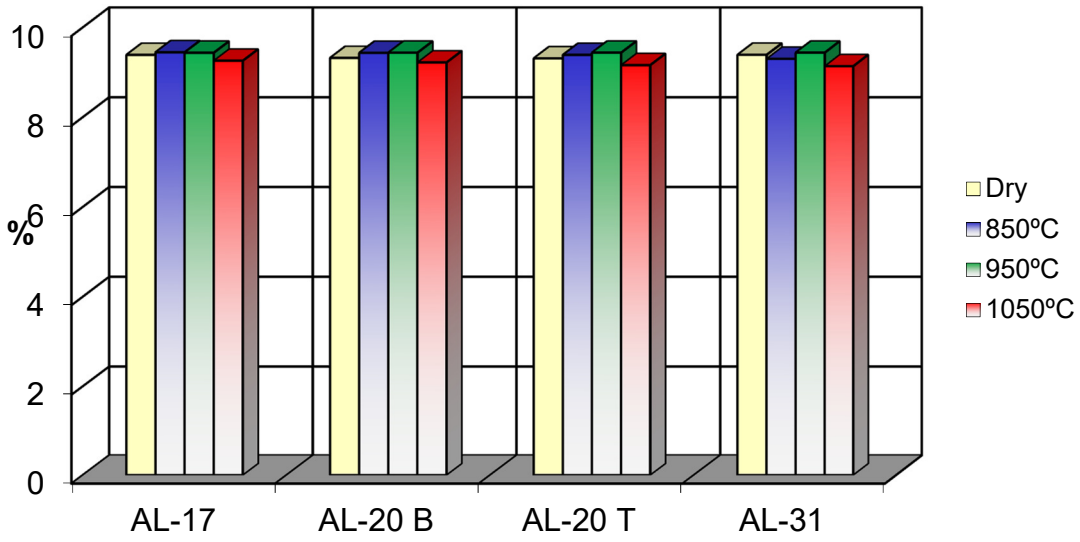

Figure V. 5 The column shows values that correspond to the total retraction values of different temperatures.

The results of testing retraction of the four sample pieces shown in column of dry (110 ° C) and firing (850 ° C, 950 ° C, 1050 ° C) results in almost the same retraction value or different variations in values no more than 1%, which ranges between 9,3% - 9,5%.

From these results and retraction comparisons were made in the drying sample and the firing sample there was no significant change in the retraction value, because the drying process of the sample was dehydrated. So for the retraction of the four samples the drying process is very important.

V.4.2 Result of Mechanical Flexural Strength (MFS)

Test MFS on sample pieces after dry and firing with different temperatures (Figure V.6) to assess the compressive strength capacity of ceramic raw materials when experiencing a pressure or impact.

Figure V. 6 MFS testing process on sample pieces at different temperatures (dried and after firing).

The graphic below shows the values corresponding to the mechanical flexural strength (MFS), for each sample, dry and after firing of the specimens with different temperatures (Figure V.7).

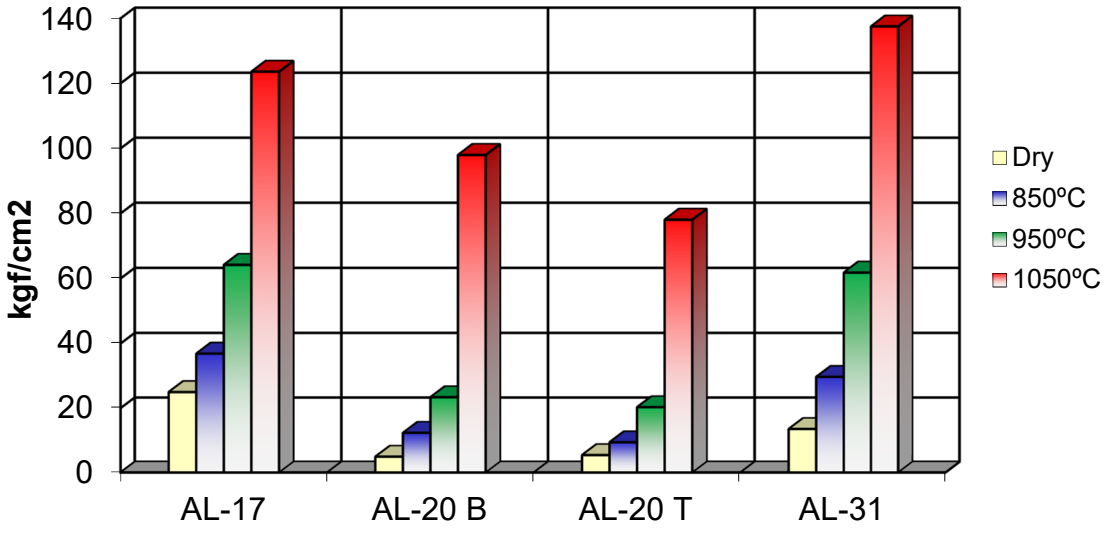

Figure V. 7 The column shows values that correspond to the total MFS values of different temperatures.

V.4.3 Result of water absorption

Water absorption test on sample pieces from different temperatures to determine the ability of water absorption in ceramic raw materials in the final product (Figure V.8).

Figure V. 8 The process of firing sample pieces at three different temperatures.

The graphic below (figure V.9) shows the results of water absorption from 3 temperatures (850°C -950º - 1050°C).

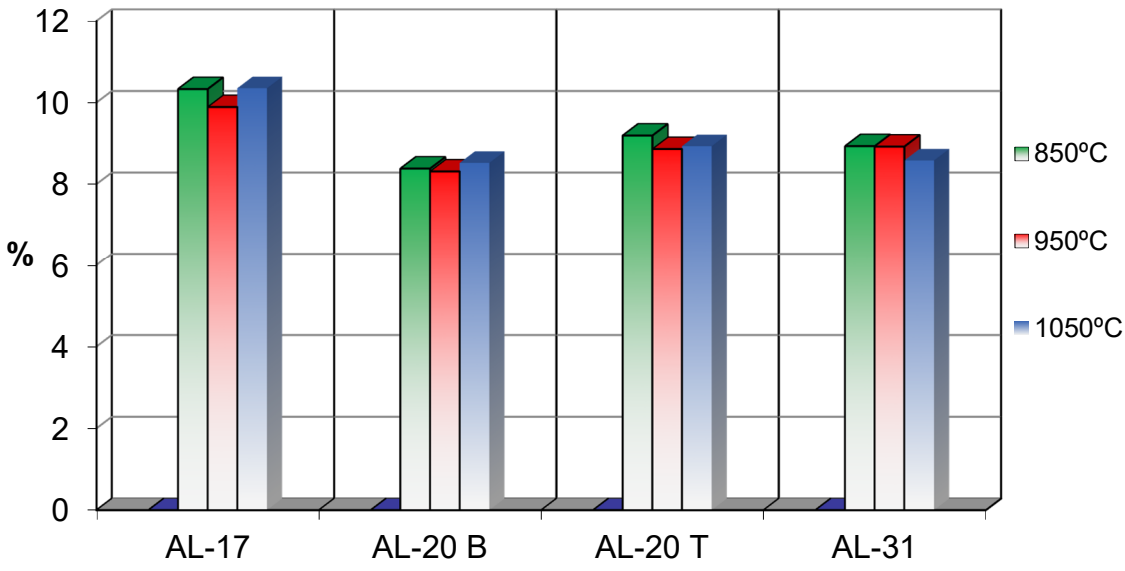

Figure V. 9 Column show the value water absorption with different temperature.

The percentage of water absorption is influenced by porosity, permeability, and grain size distribution. Small porosity, the absorption of water will be low, and vice versa large porosity, the absorption of water will be high. Permeability is seen in the particle size distribution, if the clay dominates the water absorption will be large, and if the clay is small then the water absorption will be low, this is seen in the grain size distribution of the AL-17 sample showing slightly higher water absorption, possibly as a result of a percentage smaller clay fraction (<2μm). From the results of the four samples at firing temperatures at low to high temperatures there was no significant difference, only in the AL-31 sample, water absorption was lower at higher firing temperatures, which means that porosity was slightly lower, indicating higher density. The effect of temperature on the rate of water absorption is not significant.

V.4 Whiteness degree

The determination of the degree of whiteness, carried out on a colorimeter, is intended to quickly and objectively establish the whiteness of a kaolin by comparing it with a known standard whiteness sample. A double-ray spectrophotometer with 457 nm and 750 nm wavelengths radiation sources was used in this test. Whiteness was measured on a UV-VIS spectrophotometer, fitted with an integrating sphere system using a standard barium sulphate plate as reference. % Whiteness degree is the value of transmission at 457 nm.

Table V. 4 Results percent of whiteness in the kaolin sample

Sample		Whiteness (%)		
	Powder (tablet)	Fired test pieces		
	Dry	850°C	950°C	1050°C
AL-17	38.4	33,5	35,3	30,6
AL-20B	38	32,5	32,3	32,3
AL-20T	30.4	16,7	15,5	15,6
AL-31	42.3	40,6	42,5	40,6

Whiteness degree testing is one of the most important aspects for technological applications, because one of the main characteristics intended for kaolin-based products is that they are white. The nature of a color is basically influenced by its

chemical composition, because the presence of iron oxide and titanium oxide gives brown, red or pink colors. From the results of whiteness testing, it was shown a clear relationship between the degree of whiteness of the powder material and after firing. Sample AL-31 has the highest degree of powder whiteness and after firing and sample AL-20T has the lowest degree of powder whiteness and also after firing. The degree of whiteness is related to the iron oxide content. The highest whiteness AL-31 sample has the lowest iron oxide content, and the AL-20T sample has the lowest whiteness as a result of its higher iron oxide content (Figure V. 4).

V.5 Efflorescences

The results of color and efflorescences by firing sample pieces with different temperatures (850°C, 950°C, and 1050°C) are shown in (Figure V.8). From the picture, it shows the change of color and efflorescences on the sample pieces. After that the sample pieces are analyzed to determine the percentage of whiteness and efflorescences for each different temperature.

The results of the efflorescence is shown in the picture above (Figure V.10) after firing with different temperatures show that the sample contained efflorescences on the sample pieces. Of the four samples from AL-17 at temperatures of 850 ° C and 1050 ° C, efflorescence did not occur, but at temperatures of 950 ° C efflorescence occurred which were shown in greenish yellow. The AL-20B and AL-20T samples did not show efflorescences. For the AL-31, the sample pieces at temperatures of 850 °C and 1050 °C show slight efflorescences, but at temperatures of 950 °C there are more obvious efflorescences with a greenish yellow color.

Figure V. 10 Results of color and efflorescence after drying and firing the sample pieces at different temperatures.

V.6 Resource Estimation

To estimate the potential resources occurring in the three areas of influence of the tested samples, the respective areas and thickness of the clay bodies observed in the field were considered, and a density of 2 has been considered. According to the

following table V.5 the kaolinitic clay resources occurring in the Aileu zone are estimated to be over 18 x 10^6 tonnes.

Table V.5 Resources estimation

Sample areas representative	Areas	Thickness (m)	Volume (m^3)	Density (kg/m^3)	Ton (m^3)	Total Ton (m^3)
AL-17	0,9 km^2	3 m	2.700.000 m^3	2	5,4 x 10^6	**18,3 x 10^6**
	90 ha					
	900.000 m^2					
AL-20B	0,9 km^2	>3 m	>2.700.000 m^3	2	>8,1 x 10^6	
	90 ha					
	900.000 m^2					
AL-20T	0,9 km^2	2 m	100.000 m^3	2	3,6 x 10^6	
	90 ha					
	900.000 m^2					
AL-31	0,3 km^2	2 m	600.000 m^3	2	1,2 x 10^6	
	30 ha					
	300.000 m^2					

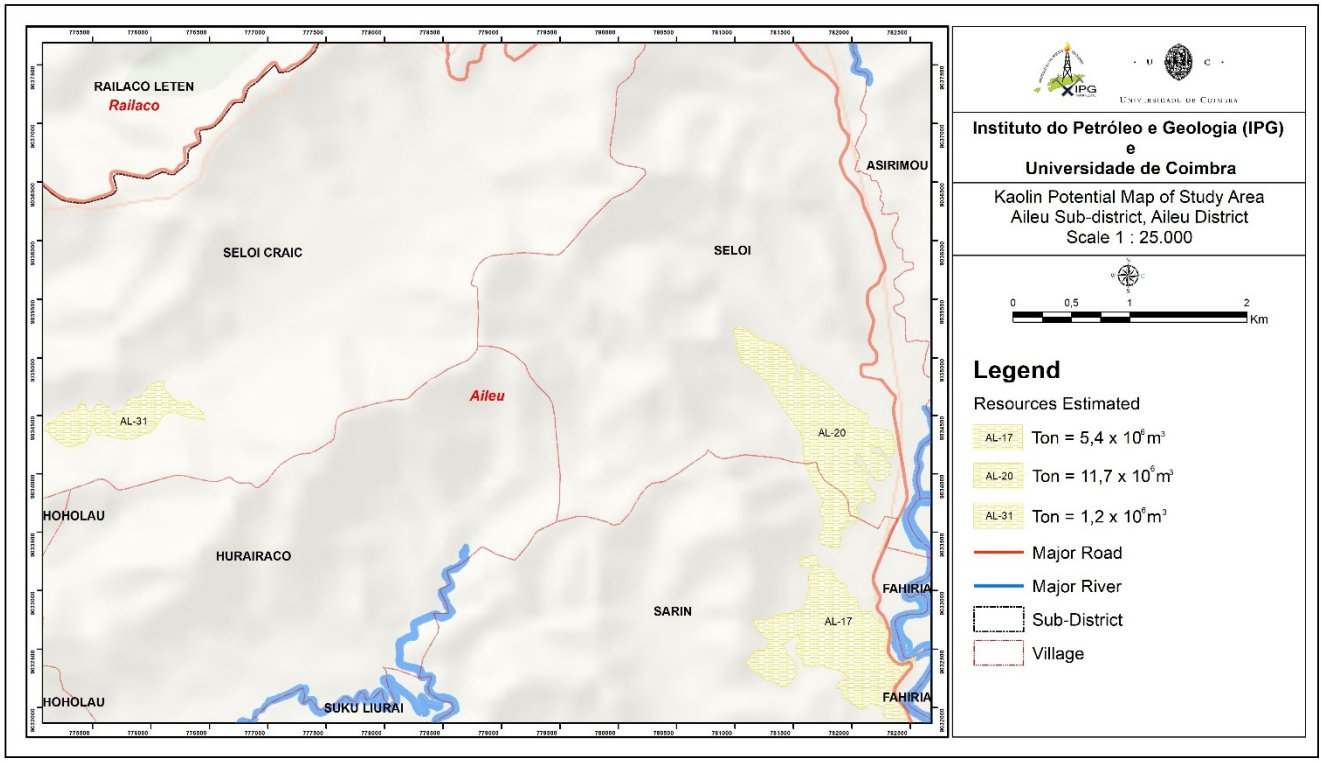

Figure V. 11 Kaolin potential map in study area, Aileu sub-district, Aileu District

CHAPTER VI. CONCLUSION

Based on the results of field studies and laboratory studies, it can be concluded as follows:

The study area is administratively located in the Aileu district. Based on regional geology (Audley Charles 1968) the stratigraphic study area can divided into two formations namely the Aileu formation (Permian age) and the Ainaro gravel formation (quaternary age). After a geological study with a scale of 1: 25 000, the study area can be divided stratigraphically into 4 units, namely: mudstone low alteration unit, mudstone high alteration units, gravel quaternary units, kaolin units and alluvial deposits. The kaolin units have very varying thickness ranges from 20 cm - 3 m, and they are always intercalated with gravel, and they show different color characteristics (white, yellowish white, reddish, gray) which are strongly influenced by different mineral and chemical compositions.

The results of field work show that kaolin contained in this area occurs through the hydrothermal alteration process (meteoric water), weathering the two processes as a primary occurrence and sedimentary processes as a secondary occurrence. The main rock of the alteration is the mudstone high alteration. Evidence of alteration and weathering is the presence of quartz veins, parts of the fracture of the host rock filled with hot liquid. The most intensive weathering occurring on the surface of the source rock which has been partially transformed into kaolin. Beside that supported by mineralogical analysis data, it shows the presence of mineral clays (kaolin / illite) and goethite which occur as a result of alterations in weathering and hydrothermal environments.

Although the raw material of the sample is not very plastic, since it is mostly silto-kaolinitic, it has iron contents that significantly affect its color, which varies from cream to reddish tones. This makes its use as a filler and coatings unfeasible (white ceramic, paper, paint industry, rubber, cosmetics and others).The best use of these kaolins is in the red structural ceramics, essentially brick, but it still requires mixing with other montmorillonite / illitic clay and sand to correct some of the revealed technological parameters, in particular retraction and mechanical strength, in order to optimize a ceramic paste for industrial use. Another area in which the use of this

kaolin could prove important is in the elaboration of building blocks with raw earth. This very easy-to-produce millennial construction, common in many areas of the world but not in the Asian region where Timor is located, and it could allow for the construction of cheap building blocks with local raw materials, and significantly improve living conditions in rural areas of the country.

Recommendation

The kaolinitic clays of Aileu constitute an important geological resource of East Timor. Considering that its industrial applicability will be very directed towards the red-base structural ceramics, it would be advisable to continue geological studies in places near Aileu, but in formations derived from basic and ultrabasic rocks, in order to identify other clays with a montmorillonite/illitic typology capable of formulating a paste optimized for the production of quality ceramic products.

References

Audley-Charles, M. G. (1968). The geology of Portuguese Timor: Memoirs of the Geological Society of London, v. 4.

Alves, R. I., Sampaio, C. F., Nadal, M., Schuhmacher, M., Domingo, J. L., & Segura-Muñoz, S. I. (2014). Metal concentrations in surface water and sediments from Pardo River, Brazil: human health risks. Environmental research, 133, 149-155.

Berry, R. F., & McDougall, I. (1986). Interpretation of 40Ar/39Ar and K/Ar dating evidence from the Aileu formation, East Timor, Indonesia. Chemical Geology: Isotope Geoscience Section, 59, 43-58.Standley, C. E., & Harris, R. (2009). Tectonic evolution of forearc nappes of the active Banda arc-continent collision: Origin, age, metamorphic history and structure of the Lolotoi Complex, East Timor. Tectonophysics, 479(1-2), 66-94.

Bloodworth, A. J., Highley, D. E., & Mitchell, C. J. (1993). Industrial minerals laboratory manual: kaolin.

de Souza Santos, P. (1975). Tecnologia de argilas aplicada às argilas brasileiras: Aplicaçoes (Vol. 2). Editora E. Blücher.

Grim, R. E. (1968). Clay mineralogy, 2nd ed., McGraw-Hill, 596p.

Gooseff, M. N., McKnight, D. M., Lyons, W. B., & Blum, A. E. (2002). Weathering reactions and hyporheic exchange controls on stream water chemistry in a glacial meltwater stream in the McMurdo Dry Valleys. Water Resources Research, 38(12), 15-1.

Kirsch, A. A., & Fuchs, N. A. (1968). Studies on fibrous aerosol filters—iii diffusional deposition of aerosols in fibrous filters. Annals of Occupational Hygiene, 11(4), 299-304.

Pettijohn, F. J., Potter, P. E., & Siever, R. (2012). Sand and sandstone. Springer Science & Business Media.

Santos, P. S., & Navajas, R. (1976). Ensaios Preliminares de Piroexpansão de Vermiculita da Bahia em Forno Piloto Experimental. Rev. Bras. de Tecnologia, 7, 415-431.

Tsao, H. K., & Koch, D. L. (1997). Observations of high Reynolds number bubbles interacting with a rigid wall. Physics of Fluids, 9(1), 44-56.