

Genesis Exploration of White Clays Deposited in Thimi Area of Kathmandu Valley

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Abstract

The geological genesis of the Thimi-Sanothimi white clay deposits of Kathmandu Valley was explored employing mineralogical and chemical analyses in this research. For the study, two sample specimens of the clay having two types of particle size, i.e., about $< 63 \mu\text{m}$ (bulk) and $< 2 \mu\text{m}$ (fine), were separated with the help of standard sieve mesh which further modified using high-temperature heat, and 1 M HCl and 1 M KCl solution treatments. For the most part, both the clay fractions constituted of 2:1 type of vermiculite and mica (mainly of K-mica type) clay minerals with different feldspars and quartz phases as clay admixtures from the results of the mineralogical phase analysis. The chemical constituents of the clay particles with $< 63 \mu\text{m}$, $< 2 \mu\text{m}$, and the HCl-treated sample specimens confirmed the existence of a high quantity of SiO_2 with comparatively low Al_2O_3 which indicates the presence of fewer amounts of the vermiculite and mica minerals comparatively with feldspars and quartz admixtures in the analyzed white clay samples. Moreover, the clay sample contained a considerable quantity of Fe_2O_3 and MgO chemicals which are not favorable raw material constituents for good quality porcelains and white-ware products without refining and chemical-modifications. The present work could be the first step to explore its potentiality in various industrial sectors like table-ware, sanitary-ware, brick and tile ceramics, paper and pulp, petrochemical, pharmaceutical including environmental pollution controlling agents in Nepal.

Keywords: Elemental analysis, FTIR, K-mica, vermiculite mineral, XRPD

Introduction

Clays and clay minerals, originated from the Earth's surface rocks are formed [1], mostly by chemical weathering and/or hydrothermal activities for thousands of years [2]. They are inorganic materials having less than $2 \mu\text{m}$ in equivalent spherical diameter particle size according to the mineralogists, although soil scientists and engineers define them as the earthen materials with $< 4 \mu\text{m}$ particle size without any significances about its mineralogical phases [3]. Besides, joint nomenclature committees (JNCs) of the Association Internationale Pour l' Etude des Argiles (AIPEA) and the Clay Minerals Society (CMS) defines the clay minerals as a class of hydrated phyllosilicates forming the fine-grained fraction of rocks, sediments which impart plasticity and harden

when fired [4]. According to this definition, synthetic and fine materials are not considered natural clay minerals even though they may display the properties of plasticity and hardening on drying and firing [5].

The clay minerals are classified into major four groups based on the tetrahedral silicate (T) and octahedral aluminate (O) sheets such as; kaolin, montmorillonite/smectite, mica, and chlorite [3]. In all types of clay minerals, the T and O sheets are stacked one above the other, exposing surfaces of oxygen and hydroxyl that can interact with each other by hydrogen bonding between the layers [6]. Details about these four groups of the inorganic clay minerals, their special properties, and applications are described elsewhere [7-12]. The clay minerals are continuously used since pre-historic times [13], depending on their basic properties of

the physical, chemical, mineralogical compositions, and the structural so on [14,15], because it becomes one of the fascinating topics for the mineralogists, industrialists, and the business entrepreneurs.

The phase composition, structure with large specific surface areas of the fine-sized clays results in distinct properties, which makes them effective properties to remove hazardous metals from the contaminated environments. Nowadays, natural clay minerals act as cost-effective natural scavengers for environmental pollutant remedies [16-20]. Moreover, the naturally deposited inorganic clays and clay minerals are practiced to use for the treatment of bacterial diseases from the earliest recorded history to recent years [21,22]. The uses of these natural clays in various industrial sectors and purposes depend mainly on their types and properties. In particular, all types of clay properties are affected mostly by their mineralogical phases, structures, and compositional structures which are frequently differ/deviate from their ideal compositional formula, because of isomorphous substitution (e.g., Al^{3+} for Si^{4+} in the tetrahedral sheet and Mg^{2+}/Fe^{2+} for Al^{3+} in the octahedral sheet) in the layered-aluminosilicates structure of the clays and such substitution enhanced almost all kinds of their properties. Hence, it is necessary to characterize and carry out the rational analysis of such naturally deposited all types of clay minerals for identifying their conclusive possibilities in industrial sectors and for diverse uses [23].

Quantitative determination of mineralogical phases present in the clay minerals by combining quantitative chemical composition and qualitative analysis is known as rational (fractional) mineralogical analysis. The main purpose of the rational analysis of the clay minerals is to determine the rational composition, percentage of the different mineral compounds such as kaolin, montmorillonite/smectite, mica and chlorite group minerals, quartz, feldspars so on. It gives us a much better conception of the true character of the clay materials, although the process of rational analysis and calculation becomes much more complex. The rational analysis is furthermore useful only in connection with mixtures of high-grade clays, in which the variation of the ingredients can only be within comparatively narrow limits. It allows us for the prediction of the physicochemical and all possible technological properties of the clay minerals. For the purposes, both the qualitative and quantitative examination is frequently carried out to acquire the

proportional amounts of clay minerals in both the bulk rock and the clay fractions. For the purposes, X-ray powder diffraction (XRPD) and Fourier transform infra-red (FTIR) techniques for the qualitative and the chemical element analysis for the quantitative analysis are frequently applied [24].

In the past, a few researchers studied the geological genesis [25-29], mineralogical [30-33], physico-sintering [34-37], and ceramics [38-40] properties of clay minerals/soils of Nepal. A detailed study about the properties of porcelain raw materials deposited in Pachamane (Shivapuri conservation area of Kathmandu district) and Daman (Makawanpur district) areas of Nepal was carried out since the 1990s [9, 32, 41-43]. However, there are no detailed research works carried out yet on the geological genesis study of the natural white clay minerals deposited in the Thimi area of Bhaktapur, except their corrosive behavior to the buried-metallic pipelines [44,45], and adsorption characteristics [18]. In this context, the present work aimed to explore the geological genesis of the Thimi white clays based on their qualitative and quantitative data analyses. This study would be very useful for further explorations of other clay deposits in ceramic and other relevant industrial sectors in Nepal.

Materials and Methods

Description of sampling site

Kathmandu Valley looks like a shallow basin surrounded by Shivapuri, Nagarjun, Chandragiri, Pulchowki, and Nagarkot, which has typical lacustrine sediments which have attracted many geoscientists. Such sedimentary soil studies were mostly established on the recorded borehole data for diverse purposes [46]. The studies faced several important problems of stratigraphic division and nomenclature of the formations, mainly because of lack of information on the clay mineralogy of the surface to subsurface and insufficient description on the definition of each formation of Kathmandu valley. Only a few researchers have carried out the lithological and mineralogical evaluation of Kathmandu valley soils [30,31]. The Paleo-Kathmandu Lake is likely to have been initiated at around 2.1 Ma and to have been filled with black organic mud, the *Kalimati* clay [47]. However, lacks detailed studies carried out on the geo-mineralogy of white clay deposited between Arniko Highway and Sanothimi-Bhaktapur roadways areas of Bhaktapur district within Kathmandu valley of Nepal. The clay sample which is called "*Kamero Mato*" in Nepali means white soil and '*Takucha*' in Newari language

[8] occurs as a deposit over wide areas of the sampling site, and it is located between 27°40'30"–27°40'50" N latitude and 85°23'40"–85°24'20" E longitude (Fig. 1). About two kg of clay sample that represents more or less all the clay deposited areas of the sampling site was collected to conduct this research work.

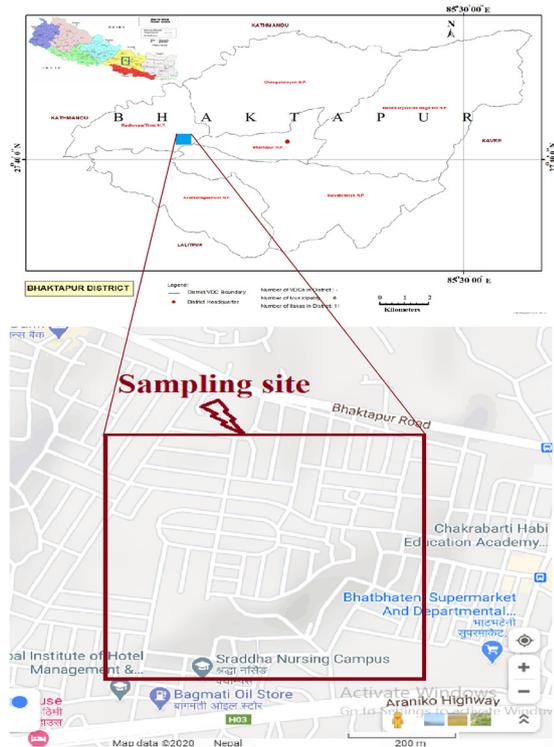


Figure 1: Clay sampling site located between Sanothimi-Bhaktapur and Araniko Highway areas of Thimi

Clay sample preparation

The bulk sample (BCS) was prepared by using a US standard sieve, mesh no. 230 for the separation of < 63 μm particles. The organic substance in the separated clay sample was treated with excess H_2O_2 (30 % by volume) on a boiling water bath until effervescence ceased completely. The process was repeated 2/3 times, washed with distilled water, and dried overnight on a hot oven at 110° C for obtaining the bulk clay particles. Moreover, fine clay minerals having < 2 μm particles were separated using the elutriation process based on Stoke's law using "Calgon" as described elsewhere [3,9] which is named as fine clay particles (FCP) hereafter. Heat treatment at different firing temperatures and chemical treatment using 1 M HCl and 1 M KCl solutions were accomplished for both the bulk and the fine clay samples.

Chemico-mineralogical analysis of clay sample

The chemical compositions of the bulk and the fine

clay samples as well as the chemically treated fine clay samples were analyzed using the X-ray fluorescent method for SiO_2 , atomic absorption spectrophotometric method for Fe_2O_3 and Al_2O_3 , EDTA-titration method for CaO and MgO, a flame photometric method for K_2O and Na_2O and gravimetric method for ignition loss at 1000° C. similarly, mineralogical phases and functional group identification present in the clay minerals were done using the recorded XRPD and the FTIR data, respectively. AD8 Advance Diffractometer (Bruker, Germany) with $\text{CuK}\alpha$ radiation ($\lambda=0.15418$ nm) at a scanning speed of 2°/min in 2 θ mode for recording XRPD patterns, and an IR Prestige-21 FTIR Spectrometer (Shimadzu, Japan) were used for such purposes. The identification of the clay minerals was made based on their basal spacing using the Joint Committee for Powder Diffraction Standards (JCPDS) files of clay minerals [48].

Results and Discussion

Chemical analysis

Table 1 shows the estimated chemical compositions of the bulk and fine clays [39], and HCl treated fine clay samples. The amounts of SiO_2 , Fe_2O_3 , Na_2O , CaO are considerably higher in the bulk clay sample as compare in both the fine clay samples without and with 1 M HCl treatment, while K_2O , Al_2O_3 , and MgO are lower in the bulk sample than in fine and HCl treated clay samples. The decrease of SiO_2 amount with the increase of Al_2O_3 in the fine clay sample than in the bulk indicating that the amount of clay minerals in the fine clay sample is more than in the bulk sample [39]. Similarly, the high amounts of MgO and K_2O are found in the fine clay specimen than in the specimen with < 63 μm particle size. This implies that the Kamerotar clay mineral with < 2 μm average particle size constitutes mainly of SiO_2 and Al_2O_3 with a considerable amount of MgO. A considerable amount of MgO with more than 1 %, i.e., 1.86 % Fe_2O_3 in the fine clay minerals of the Kamerotar clay is not a suitable raw material for the industrial production of good quality porcelains or white wares without modifications and treatments. For example, the HCl treatment method might be best among others to remove the Fe_2O_3 from the sample, because Fe_2O_3 decreased with 1 M HCl treatment, as shown in Table 1. It is worthwhile to cite the certainty that a substantial amount of Fe_2O_3 and K_2O in clay minerals ascribed to be iron-bearing muscovite, corresponding to K-mica [3].

Table 1: Chemical composition of the Thimi white clay specimens of Bhaktapur, Nepal

Oxides	Weight (%)		
	Bulk**	Fine**	HCl-treated
SiO ₂	66.50	60.18	61.33
Al ₂ O ₃	14.42	15.95	14.99
Fe ₂ O ₃	3.86	1.86	1.58
MgO	2.01	8.43	8.30
K ₂ O	2.16	2.64	2.43
Na ₂ O	0.90	0.45	0.46
CaO	0.56	0.24	0.19
LOI*	7.83	9.96	10.04
Misc.	1.76	0.29	0.68

* at 1000° C, ** ref [39]

Mineralogical phase analysis

The XRPD analysis of clay minerals and ceramics is one of the effective methods for their mineralogical phase identification studies, although some circumstances make complications for precise phase identification from overlapping the peak values [49, 50]. Figure 2 shows the XRD patterns of the bulk, fine, and HCl treated clay samples. The reflection peaks at 1.449 nm, 1.00 nm, 0.72 nm, 0.290 nm, 0.256 nm, 0.199 nm, and 0.166 nm are assigned for vermiculite clay mineral which is observed in both the bulk and fine clay samples, as shown in Fig. 2.

The XRPD peaks at 1.00 nm, 0.720 nm, 0.256, 0.199 nm, and 0.166 nm are more clearly observed in the fine clay sample than in the bulk clay sample. In contrast, the peak intensity at 1.449 nm for the fine clay sample is low as that of the bulk clay sample. Besides, the K₂O content greater than 0.35 weight % [51] in all analyzed clay samples suggests that Kamerotar clay is not pure vermiculite. It might contain mica, mica/vermiculite interstratification that is in agreement with the XRPD results also (Fig. 2).

The presence of mica mineral in both bulk and fine clay samples is indicated by its 001 basal spacing at 1.00 nm with the second-order basal spacing (002) at 0.496 nm. Apart from these, the peaks at 0.448 nm, 0.334 nm, and 0.256 nm are also assigned for mica minerals. Furthermore, it is important to identify whether the mica mineral is of muscovite (K-mica) or paragonite (Na-mica) type mineral. The peaks at 1.00 nm and 0.496 nm (i.e., not at 0.96 nm and 0.48 nm) indicate that the Kamerotar clay sample contains mostly of a muscovite type of mica minerals. The same method of the identification of K-mica or Na-mica mineral was also used previously [52].

Strong reflection peaks at 0.427 nm, 0.334 nm, 0.246 nm, 0.228 nm, 0.213 nm, and 0.182 nm are assigned for quartz which is not a clay mineral

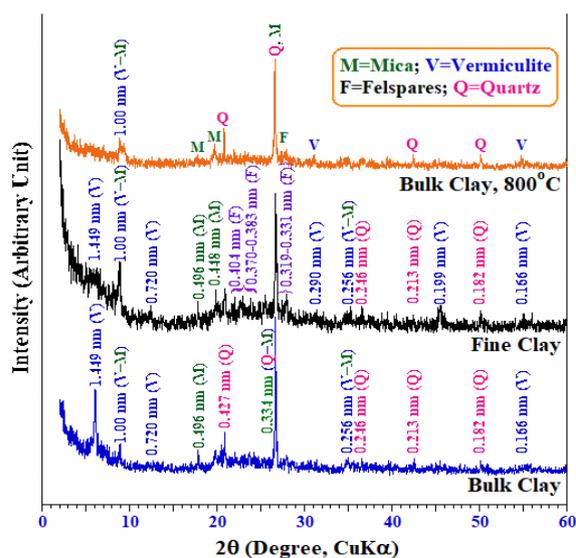


Figure 2: XRD patterns of the bulk clay, fine clay, and the bulk clay sample heated at 800°C

although frequently present in < 2 μm clay fraction. The presence of quartz and mica in the clay samples might be the reason for showing a strong XRPD peak at 0.334 nm. The 0.331 nm peak for mica minerals is generally overlapped with the quartz peak. The 2nd most intense peak for the quartz is confirmed from the 0.427 nm. The intensity of the second most intense reflection peak at 0.427 nm for the quartz is observed in the fine clay sample. Less intense reflection peaks at 0.404 nm, 0.383-0.370 nm, and 0.331-0.319 nm are attributed for feldspars, mostly of potassium-feldspar which is also supported by the chemical analysis data, as shown above in Table 1. More intense reflections of the feldspar in the fine clay sample than in the bulk clay are due to an increase of K₂O in fine clay particles than in bulk clay from in the chemical analysis data. Furthermore, the presence of a 1.449 nm reflection peak is due to the vermiculite or/and chlorite minerals, because both these clay minerals have the basal reflection peak at around 1.4-1.5 nm. The vermiculite clay mineral is usually identified based on its strong peak at 1.449-1.40 nm, which collapsed to 1.00 nm after heat treatment of the bulk clay sample at 600°-800° C for two hours, and the peak at 0.720 nm is also collapsed for the bulk clay sample (Figs. 2 and 3). It demonstrates that the interlayer OH⁻ of vermiculite should be destroyed [53].

A similar effect of the complete collapsing of XRPD peaks at 1.445 and 0.720 nm in the bulk clay samples treated with 1 M KCl solutions, as shown in Fig. 4. The similar effects of KCl and heat treatments were

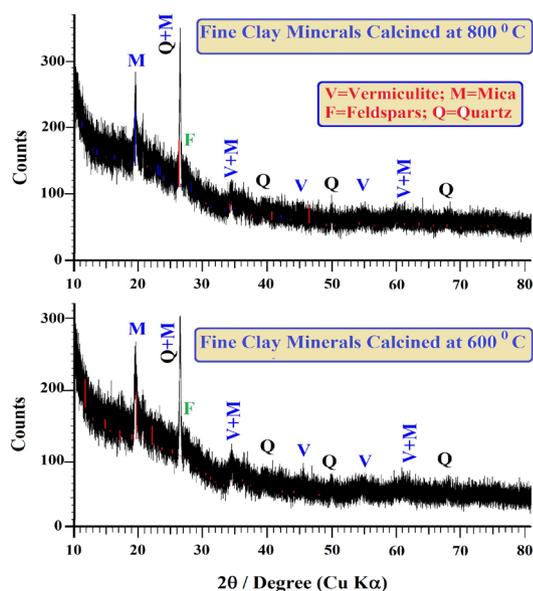


Figure 3: XRD patterns of the fine clay sample after heat treatment at 600°C, 800°C for 2 hours, and without the treatment for comparison

reported in previous studies for differentiating between vermiculite and chlorite minerals [54]. It reported that the collapse of the 1.45 nm basal spacing on heat treatment and relatively low intensity of the second-order basal spacing at 0.72 nm pointed to a structure

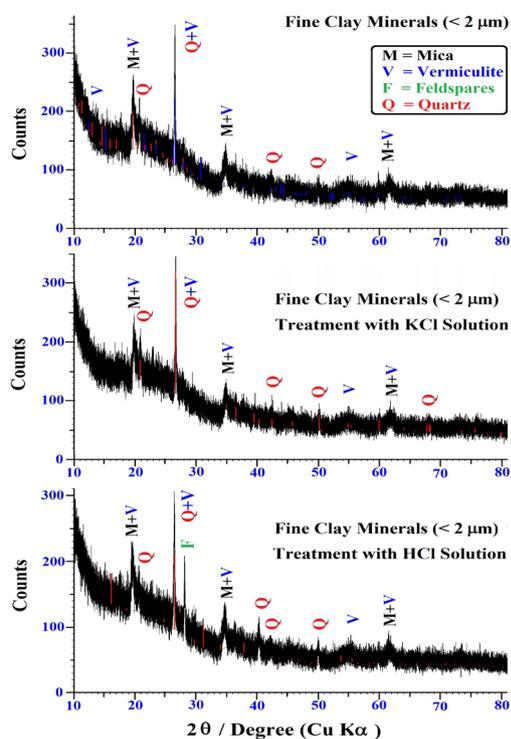


Figure 4: XRD patterns of the fine clay sample after chemical treatment in HCl, KCl solutions, and without the treatment for comparison

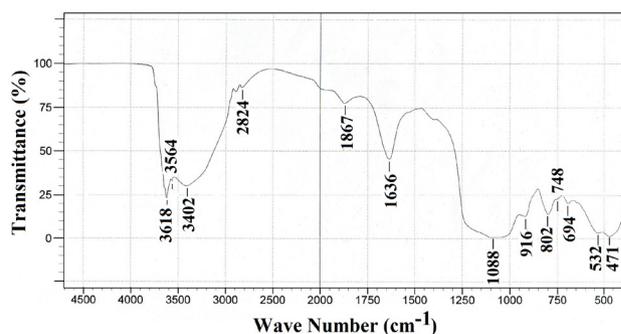


Figure 5: FTIR spectrum of the fine clay sample of the Kamerotar area

more closely linked to vermiculite clay mineral than to the chlorite type mineral [55]. Consequently, the clay sample of the Kamerotar area consists of vermiculite clay minerals, not chlorite type. It is noteworthy to mention here that the muscovite type mica (i.e., K-mica) with paragonite (i.e., Na-mica) observed in single mica assemblages in middle-grade metamorphic rock, for which it reported the highest Na/(Na+K) ratio, i.e., 0.31 [52]. However, the ratios of Na/(Na+K) in both the bulk and fine clay samples were found to be 0.29 and 0.20, respectively. These results revealed that the mica in the fine clay of Kamerotar is of muscovite. Consequently, major clay minerals identified in the Kamerotar clay sample of Bhaktapur, Nepal, are vermiculites, muscovite type of mica with feldspars and quartz as clay assessors chemical components from XRPD analysis.

FTIR spectroscopy is also one of the noted techniques for the confirmation of the mineralogical phases present in the clay samples, and it enables us to obtain sufficient information about individual minerals, non-crystalline admixtures, so on. It makes it possible to distinguish the different components of clay minerals [56,57] and influences by a degree of crystalline order [58] and particle size of the natural clay materials [59]. Three bands between 3800-3000 cm^{-1} regions are observed for the fine clay (Fig. 5). A strong band at 3618 cm^{-1} attributed to Al–OH–Al with Al–Mg–O stretching vibrations. The existence of an interlayer –OH group between tetrahedral and octahedral sheets of the clay minerals gives an FTIR band near 3620 cm^{-1} [60]. Therefore, the FTIR peak at 3618 cm^{-1} for both the fine clay minerals associated with the inner hydroxyl group present in the clay samples of Kamerotar. The peak at 3564 cm^{-1} is mostly of the FeFeOH grouping in the octahedral sheets of the minerals that are also consistent with previous reports [61]. Besides, the broad peak near 3400 cm^{-1} indicated

the stretching H–O–H vibrations [59], and hence the broad peak at 3402 cm^{-1} is considered to interlayer hydroxide in Kamerotar fine clay sample.

It is meaningful to cite here that the vermiculites showed the –OH bending vibration mode at 1635 cm^{-1} [56] that is in good agreement with the band of H–O–H stretching vibration of the present clay sample. Consequently, it can sum up that the Kamerotar white clay consists of vermiculite type clay minerals. Furthermore, only one broad and complex Si–O stretching band at 1088 cm^{-1} is observed in the FTIR spectrum of the fine clay sample, and it might be because quartz was also assigned to the Si–O stretching at 1080 cm^{-1} [62]. The absorption bands at 802 cm^{-1} , 748 cm^{-1} , and 471 cm^{-1} are due to the bending vibration of Si–O–Si linkage in quartz [63]. These results are consistent with the XRPD analysis (Fig. 2), as described above. FTIR bands near 694 cm^{-1} 532 cm^{-1} are due to the Al–O–Si and the Mg–O linkages, respectively. The Al_2O bending band of the clay minerals near 918 cm^{-1} arises from vibrations of the inner –OH group of the clay minerals [64].

Conclusions

Major clay minerals identified in the Thimi white clays are vermiculite, micas, feldspars, and quartz-based on the chemical, XRD, and FTIR analyses. Considerable amounts of Fe_2O_3 and K_2O present in the clay sample is attributed to be iron-bearing muscovite, analogous to K-mica. Furthermore, the considerable amounts of Fe_2O_3 and MgO contained in both the fine and bulk clay samples compared to the standard clay raw materials used for the porcelain productions indicated that these clay deposits are not suitable raw materials for a good quality of the white-ware productions without modifications and purification, for example, HCl treatments. Instead, it may be used as a suitable adsorbent for removable of toxic ions or compounds from contaminated water.

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