

## Research Article

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## The Development of Pigments Reflects Infrared Radiation from Gravel (Lateritic) Soils

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### Abstract

This research aims to analyze pigments from lateritic soil to reflect infrared radiation. The lateritic soils used in this research were from Ban Wanghai, Mukdahan Thailand. The study started by analyzing the physical properties and chemical composition of the soil to develop the NIR reflective pigments. The lateritic soils were ground and reduced to sizes smaller than 45 millimeters, then were calcined at 900-1300°C for 1 hour. The calcined pigments were characterized and observed by XRD and SEM, and infrared reflection was measured by use of a UV-Vis-NIR spectrophotometer. The results of the research have shown that highly reflective NIR pigments from the soil reflect about 70-80% of infrared radiation at 1000°C calcined firing temperature. It was found that quartz and FeAl<sub>2</sub>O<sub>4</sub> were the main constituents. The particle size shows that the highest NIR reflectance is around 1-20 micrometers.

**Keywords:** NIR reflective pigments, Calcined, Lateritic soil

### 1. Introduction

Infrared radiation makes up approximately 50 percent of the Sun's radiation that reaches Earth and is responsible for heating the planet. This is because infrared radiation excites the molecules, vibrating them and changing the position of the atoms, causing the substance to heat up (1, 2). Infrared radiation can be divided into 3 ranges: 1) IR-A, 2) IR-B, and 3) IR-C. IR-A is the majority of the radiation from the sun that reaches the Earth. IR-A has been reported to have a biological effect on human skin. When IR-A comes in contact with the skin, it will be absorbed into the skin causing the skin to lose elasticity and reduce skin firmness, resulting in shortened skin life (3). Infrared radiation can cause human skin to have a temperature higher than 40°C (4). Skin that is exposed to infrared radiation for a long period of

time can accumulate the radiation and has adverse effects on the skin, including deep wrinkles, aging skin, lost skin elasticity, skin dullness, freckles or dark spots, and the most frightening being skin cancer. Farmers and construction workers who have to work in the sun are at a higher risk of the conditions mentioned above.

There is a community at Ban Wanghai, Phu Wong Sub-district, Nong Sung District, Mukdahan Province who use red clay or laterite soil to dye their own clothes, red robes offered to meditation monks, and other clothes for sale. The clothes that are dyed from the clay are considered their community product. If the clothes can reflect heat and prevent IR-A from affecting the skin, that could help to prevent the skin from getting damaged which will lead to having a longer life.

To solve health problems caused by exposure to large amounts of NIR radiation, researchers focused on the development of infrared reflective pigments from laterite soil which has infrared radiation type NIR or IR-A reflective properties through the main components of the laterite soils which include silica ( $\text{SiO}_2$ ), alumina ( $\text{Al}_2\text{O}_3$ ) and iron oxide ( $\text{Fe}_2\text{O}_3$ ) which are extracted by conducting size separation and calcination at various temperatures. Which are then ground into sizes bigger than half the wavelength of infrared radiation (5) to make pigments for the community to dye their fibers and weave them into fabrics capable of reflecting infrared radiation.

### Research Objectives

1. Study the physical and chemical characteristics of lateritic soils from Ban Wanghai, Nong Sung District, Mukdahan Province, Thailand.
2. Develop pigments from lateritic soils in the temperature range of 900 – 1300°C to reflect infrared radiation type NIR with solid-state reaction.

## 2. Materials and Experiment

The development of infrared reflective pigment from lateritic soils is carried out as follows:

1. Collect samples of lateritic soils from Ban Wanghai, Nong Sung District, Mukdahan Province, the source where the community brought lateritic soils to dye the fabric. These are then developed to reflect infrared radiation by grinding using a hammer mill and separated by size for particles that are able to pass through a 100-mesh sieve. After which they are ground again in a ball mill for 24 hours. Select particle sizes smaller than 45 micrometers by using a precipitation method according to Stoke's law. After which the substance is calcinated at 900-1300°C for 1 hour to form a new phase. It is because the temperature at 900°C is the pre-decomposition of hydrate and carbonate (950°C). So, the initial temperature before the decomposition of the substance was used. After that, the iron oxide phase transition temperatures were studied. Grind the substance after calcinating to sizes in the range of 1-10 micrometers.

2. The lateritic soil that has undergone the calcining process at a temperature of 900-

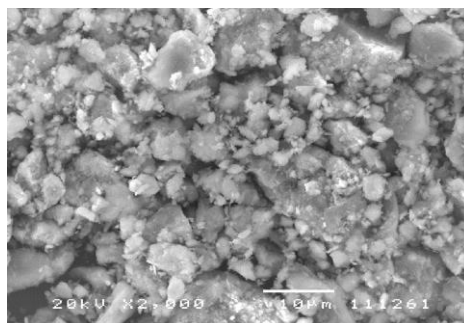
1300°C is ground to a size range of 1-10 micrometers and was analyzed by X-ray Diffractometer (Shimazu 6000, Cu target,  $\lambda = 1.54^\circ\text{A}$ , Japan) to study crystal structure, Scanning Electron Microscopy (SEM, Prisma E, Thermo Scientific, United States) to study soil particle morphology, Energy Dispersive Spectrometer (EDS-SEM, Prisma E, Thermo Scientific, United States) to find the chemical compositions, and infrared reflectance and color were measured by UV-Vis-NIR spectrophotometer (AVANTES Avalight-DHS; Detector AvaSpec-2048L). Then the distribution of particle sizes was characterized by Particle Size Analyzer (Hydro 2000s, MALVERN, England).

3. Study the effect of particle diffusion on infrared reflection on the pigment that has undergone the calcining process at a temperature of 900-1300°C. Select the temperature with the best infrared reflection. Then grind the pigment to reduce particle size by using a ring mill at 20, 40, 60, 80 and 100 minutes, respectively, and analyze the particle size and measure infrared reflectance.

## 3. Results and Discussion

### 3.1 Properties of the Lateritic Soils

The morphology of lateritic soils were shown in Figure 1, lateritic soils are agglomerate, sharp-shaped, with particle sizes in the range of 1-20 micrometers. Considering the chemical composition of lateritic soils from Mukdahan Province was analyzed by EDS as shown in Table 1, it was found that silica ( $\text{SiO}_2$ ), ferric oxide ( $\text{Fe}_2\text{O}_3$ ) and alumina ( $\text{Al}_2\text{O}_3$ ) as major compositions and titanium dioxide ( $\text{TiO}_2$ ), potassium oxide ( $\text{K}_2\text{O}$ ) and Magnesium oxide ( $\text{MgO}$ ) as minor composition and ignition loss is approximately 5.31 %.

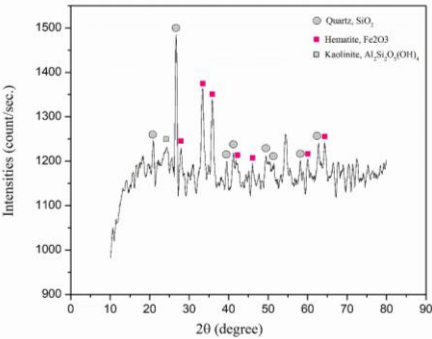


**Figure 1** SEM photograph of lateritic soils from Mukdahan Province.

**Table 1** The chemical composition of lateritic soils from Mukdahan Province.

Chemical Composition	Amount (%)
SiO <sub>2</sub>	34.94
Al <sub>2</sub> O <sub>3</sub>	13.74
Fe <sub>2</sub> O <sub>3</sub>	43.12
TiO <sub>2</sub>	1.05
K <sub>2</sub> O	1.48
MgO	0.36
Loss on ignition (LOI)	5.31
Sum	100

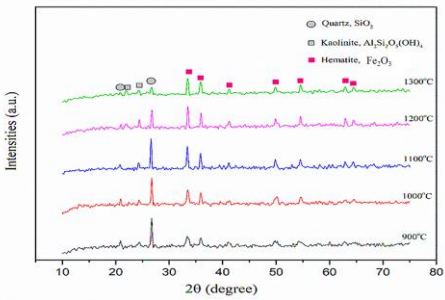
The crystal structure of lateritic soils was characterized by XRD were shown in Figure 2, the quartz phase (SiO<sub>2</sub>, JCPDS card No. 46-1045) is found which is one of the most stable forms of silica among the various forms of silica such as amorphous, tridymite and cristobalite, hematite (Fe<sub>2</sub>O<sub>3</sub>, JCPDF card No. 84-0311) is also found as called red rust and the other phase is kaolinite (Al<sub>2</sub>Si<sub>2</sub>O<sub>5</sub>(OH)<sub>4</sub>, JCPDS card No. 05-0143).



**Figure 2** SEM photograph of lateritic soils from Mukdahan Province.

**3.2 Crystal Structure of the Calcined Lateritic Soils**

The crystal structure analysis by XRD of laterite soil from Ban Wanghai. Mukdahan Province calcined at 900-1300°C as in Figure 3.



**Figure 3** XRD patterns of lateritic soils from Mukdahan Province calcined at 900-1300°C.

From Figure 3 At the calcined temperature 900°C, the quartz (SiO<sub>2</sub>, JCPDS card No. 46-1045) peaks appear highest while the hematite (Fe<sub>2</sub>O<sub>3</sub>, JCPDS card No. 84-0311) peaks are shorter. Because the organic matter in the soil obscuring the quartz peaks is burned away, XRD can better analyze the crystallinity of the quartz. When the calcining temperature was increased to 1000°C, the peak of hematite (Fe<sub>2</sub>O<sub>3</sub>) had a clearer appearance and increased. These show that higher temperatures helped to improve the arrangement of the hematite crystal structure, and crystal size increases when the temperature increases until it achieves 1300°C which calculate by the Scherrer equation ( $D=k\lambda/\beta\cos\theta$ , where  $D$  is the grain size,  $k$  is constant (0.9-1)  $\lambda$  is x-ray wavelength ( $\text{Cu} = 1.54\text{\AA}$ ),  $\beta$  is FWHM and  $\theta$  is diffraction angle) (6). The results were shown in Table 2.

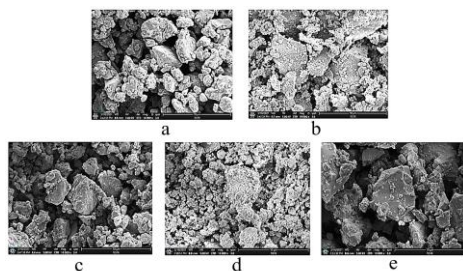
**Table 2** Crystal size of Fe<sub>2</sub>O<sub>3</sub> at the different calcined temperatures.

Calcined temperature	Crystal size
900°C	35.17 nm
1000°C	39.56 nm
1100°C	48.77 nm
1200°C	62.42 nm
1300°C	68.21 nm

The peak height of quartz as observed from the peak position at  $2\theta = 27^\circ$  will increase when increasing the temperature from 900 to 1100°C, but the peak height decreases when increasing the temperature to 1200 and 1300°C. At 1300°C, the peak height of quartz is shorter than the peak of hematite because the quartz may have reacted with kaolinite (7), but the crystalline structure has not yet formed, so it cannot be measured by XRD. Thus, at the highest temperatures, only hematite peaks are clearly detected, even though crystallinity is increased. However, it still does not transform into a spinel crystalline formation at calcining temperatures (8).

### 3.3 Morphology of calcined lateritic soils

Results of the study on the microstructure of calcined laterite soils at 900-1300°C. As in Figure 4.

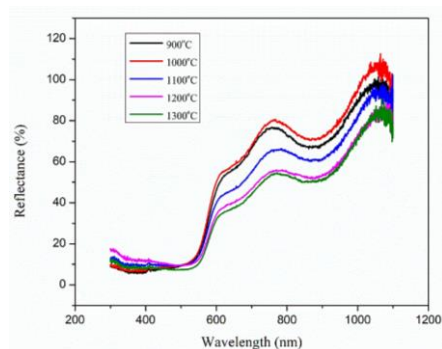


**Figure 4** Results of microstructure characterization of calcined lateritic soils at 900-1300°C, a = 900°C, b = 1000°C, c = 1100°C, d = 1200°C and e = 1300°C.

The microstructure characteristics of lateritic soils from Ban Wanghai Mukdahan Province were examined by SEM as shown in Figure 4. It was found that the calcined soil samples at 900°C as shown in Figure 4a are rough, non-rounded particles with a particle size of about 1-10 micrometers. The particles loosely stick together as large particles. The samples calcined at 1000-1300°C as shown in Figure 4b-e, the particles are sharper than those calcined at 900°C. It is because hematite ( $\text{Fe}_2\text{O}_3$ ) had a clearer appearance which is consistent with the XRD results. The particle size is in the same range of calcined at 900°C. These are particle sizes that lie at more than half the wavelength of infrared radiation.

### 3.4 Infrared reflection of calcined lateritic soils

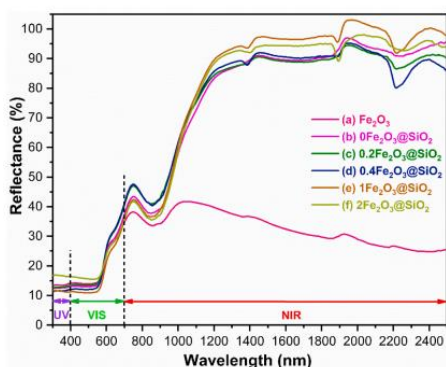
Reflection of infrared rays of lateritic soils from Mukdahan Province sintered calcine at a temperature of 900-1300°C as in Figure 5.



**Figure 5** Reflection of infrared rays of laterite soil from Mukdahan Province calcined at a temperature of 900-1300°C.

The calcined lateritic soils were measured for infrared reflectance by use of a UV-Vis-NIR spectrophotometer. The results are shown in Figure 5. Calcined lateritic soils at all temperatures or pigments have a tendency to reflect radiation in the same direction. All samples reflected low amounts of ultraviolet radiation which indicates high absorption of ultraviolet radiation. When considering visible light (wavelength range 400-700 nanometers) at wavelength range 400-550 nm, the pigments will absorb light well and reflect very low amounts of radiation (1, 2). The pigments will have a higher ability to reflect radiation in the wavelength range of 550 nanometers up until entering the infrared radiation range. With a light wavelength greater than 700 nanometers, the reflectance is higher, approaching 100%. The pigment calcined at 900°C can reflect lower infrared radiation than the pigment calcined at 1000°C due to hematite at 1000°C calcining temperature being more crystalline than hematite at 900°C (9). When the calcine sintering temperature was increased to 1100°C, 1200°C and 1300°C, infrared reflectance tended to decrease. Due to the high refractive index, crystalline quartz reacts with other substances which causes the reflecting infrared radiation to be reduced accordingly (9). There is a reduction in radiation reflection in the range of 700-1000 nanometers, it is because some of the

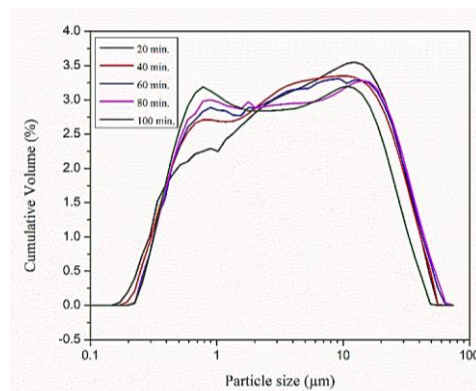
small particles contained in the pigment also cause the radiation to be scattered and absorbed by the pigment. Therefore, pigments still cannot fully reflect infrared radiation. The calcining temperature at which the pigment reflects infrared radiation best is 1000°C. When comparing the infrared reflection between calcined lateritic soils and  $\text{Fe}_2\text{O}_3$  (Figure 6) (9), it was found that the infrared reflection curves are similar. However, the calcined lateritic soils have higher reflectivity. It is because calcined lateritic soils are composed of high silica which enhances infrared reflection (9).



**Figure 6** Reflectance spectra of (a)  $\text{Fe}_2\text{O}_3$ , (b)  $0\text{Fe}_2\text{O}_3@\text{SiO}_2$ , (c)  $0.2\text{Fe}_2\text{O}_3@\text{SiO}_2$ , (d)  $0.4\text{Fe}_2\text{O}_3@\text{SiO}_2$ , (e)  $1\text{Fe}_2\text{O}_3@\text{SiO}_2$ , and (f)  $2\text{Fe}_2\text{O}_3@\text{SiO}_2$  pigments (10).

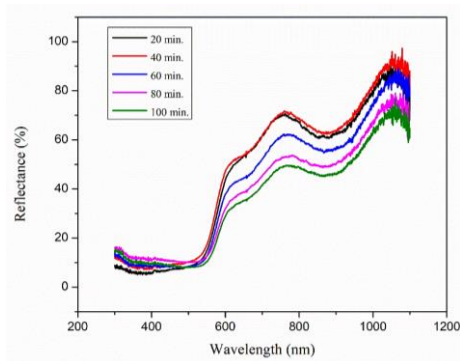
### 3.5 The effect of particle size on infrared reflection

Effect of Particle Size Distribution on Infrared Reflection calcined at 1000°C and ground to reduce particle size using ring mill at 20, 40, 60, 80 and 100 minutes respectively, then measure particle size and IR reflectivity analyzing at each time point. The result is shown in Figure 7.



**Figure 7** The pigments from lateritic soils Mukdahhan Province.

From Figure 7 The pigments from lateritic soils Mukdahhan Province were calcined at 1000°C and then ground using a ring mill at different time points. It was found that the particle size distribution was in the range of 0.2-50 micrometers. The distribution of the particle size is divided into 2 groups, at 0.5 micrometers and 10 micrometers. When increasing the grinding times, the average size in the range of the 0.5 micrometers group increased, while the average size in the range of the 10 micrometers group decreased. The limitation of the ring mill affected the distribution of the particle size. The average size of the particles ground for 20 minutes was 4.91 micrometers, 40 minutes was 4.85 micrometers, 60 minutes was 4.22 micrometers, 80 minutes was 3.94 micrometers and 100 minutes was 3.71 micrometers. The particles after being calcined consist of quartz and hematite. This makes it difficult to grind and reduce the size. That is why the size of ground particles did not differ much when increasing the grinding time. The ground particles at each time point were used to measure the reflectance of infrared radiation. The result is shown in Figure 8.



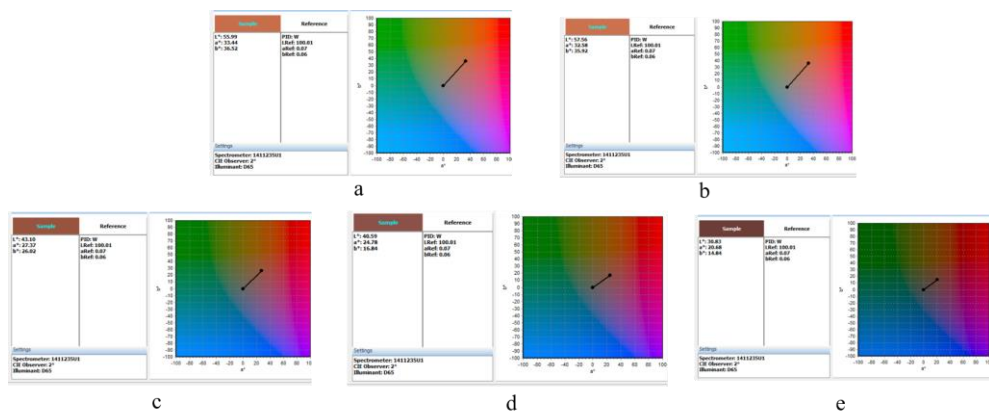
**Figure 8** Results of measurement of infrared radiation reflection of pigments from lateritic soil particles from Mukdahan province at different grinding times.

From Figure 8 The pigments from lateritic soils milled at 20 and 40 minutes had similar IR reflectivity and higher reflectivity at other grinding times. As mentioned above, there are 2 ranges of particle size distribution: the average range is about 0.5 micrometers and 10 micrometers. Small particles with less than half the wavelength of infrared radiation can absorb

infrared radiation (10). This makes the reflection lower as the particles become smaller. The wavelength of infrared radiation that strikes an object and generates heat is 700-2500 nanometers. Therefore, in order to reduce the absorption of infrared radiation as much as possible, the pigment particles should be larger than 1250 nanometers or 1.25 micrometers, which is half of 2500 nanometers. To be able to achieve the best radiation reflection, the average particle size needs to be larger than 1.25 micrometers. However, in this experiment, there are still some particle sizes smaller than 1.25 micrometers contained in the pigment, so high infrared absorption is still present.

### 3.5 The effect of calcined temperature on the color of calcined lateritic soils

Figure 9 were shown the color of calcined lateritic soils which calcined at temperatures in the range of 900-1300°C. The color of low temperature calcination is brown and became to darker and closer to black as the calcined temperature increased. It is because the iron oxide transformed from  $\text{Fe}_2\text{O}_3$  to  $\text{Fe}_3\text{O}_4$  which is black rust (7, 8).



**Figure 9** The color of calcined lateritic soils from Mukdahan Province, a. calcined at 900°C b. calcined at 1000°C, c. calcined at 1100°C, d. calcinated at 1200°C and e. calcined at 1300°C.

## 4. Conclusions

The Pigments from lateritic soils of Ban Wanghai, Nong Sung District, Mukdahan Province, have particle sizes in the range of 1- 20 micrometers. The major component of the pigment was quartz, hematite and kaolinite.

The calcining temperature that made the pigments reflect infrared the most was 1000°C. The lateritic pigments milled for 20 and 40 minutes had the best infrared reflectance, reflecting 70-80% of infrared radiation. The color of the calcined lateritic soils is brown to black.

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### Declaration of conflicting interests

The authors declared that they have no conflicts of interest in the research, authorship, and this article's publication.

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