# Red Clay Mineral Deposits: I, Prospecting, Testing and Evaluation

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### Abstract

Red clay minerals of known and unknown deposits in the provinces of Mindanao were evaluated using the direct or residue method of testing red clays for possible ceramic ware productions. The amounts of sample residue were de termined by screening one kilogram of dried clay, passing through a 74micron (200 mesh) screen. Clays containing less than 20 percent sand residue were added with river sand. Clay samples were formed into a body using the soft mud method, molding in a 22 x 6 x 1.5 cm and 2.5 x 2.5 x 4 cm metal mold, dried in the air for a week, in an oven at 110°C overnight and fired at 750°C, 850°C, 95 °C 1050°C, and 1150°C. Physical properties such as tempering water, drying shrinkage, firing shrinkage compresive strength, modulus of rupture, water absorption and porosity were measured.

Results showed that red-clay ceramic products were satisfactorily produced in clays containing more than 20 weight percent strong sand residue and in clays added with river sand. The amount of sand residue ranged from 5-41 weight per. cent, water of plasticity ranged from 21 to 33 percent, drying shrinkage ranged from 6.77 to 16.95 percent. The firing shrinkage increased with the firing temperature in almost all the samples. Compressive strength gradually increased from 750 to 850°C, decreased from 850 to 950°C and again increased at 1050°C. Water absorption decreased with increasing firing temperature. Modulus of rupture gen erally decreased above 850°C, and at 950°C some of the samples exhibited an increase in MOR while many of the samples continued to decrease in their values.

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### Introduction

Cood quality red clay minerals as well as "poor" quality red clay de-<br>Cood quality red clay bodies are clays, which can be used as single<br>body or "monobody" in the manufacture of ceramic red clay wares. Poor quality red clay bodies are clays, that cannot be directly used in the production of red clay bodies but can be mixed or formulated with river sand or quartz and the resulting body can be utilized for the production of cer tion of red clay bodies but can be mixed or formulated with river sand or objects with qualities comparable to that of good clays. In the end, all red<br>clay minerals can be utilized in the production of heavy clay wares by know-<br>ing the amount of the additives to be incorporated into the clay.

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Survey of clay resources in Region 10 reveal that 32 locations are identified and found potential raw materials for the production of building bricks, architectural terra cotta and other ceramic wares (Amable 1996). Activi Misamis by the Bureau of Mines in Bulua, Cagayan de Oro City, Naawan and Manticao<br>Misamis Oriental. (Dumaoal, et al 1982). Bulua area is now considered as<br>the ceramic center of Misamis Oriental. Sto. Nino, Catarman in the island<br>p (Ochotorena 1986). As a result, Baroy, Lanao del Norte is now considered the clay town of the province, where the Provincial Ceramics Training Center was established. Local entrepreneurs are also conducting their livelihoo by the utilization of red clays, producing jars, terra cotta, bricks and pots.

The surveys that were conducted identified the potential clay areas.

However, the deposits in the area were not fully utilized and if ever utilized the entrepreneur closed down shops due to the problem encountered by the raw materials during the actual processing. Moreover, remaining areas are to be explored so that the clay can be properly determined as to the kind of product it can be profitably produced and clays which need formulation be given right mixing proportion. As a result, ceramic livelihood industry can become more profitable business in the production not only in the traditional pots, jars and bricks but also in cheap building construction materials (roof ing tiles, building bricks) and other high end products such as stoneware bodies. This can be materialized by the cheap and abundant supply of the clay raw materials in the nearby areas.

## Methodology

#### 1. Sampling

The "cement size" sacks of clay were taken in the areas containing deposits of known and unknown deposits. In the area where there is no knowl edge of clay deposits, the barangay captain plays an important role. Coastal barangays, with friendly and cooperative barangay captains, were the first area to be prospected. A barangay visit is to be conducted and the prospec tors explain the purpose of the visit. After preliminary talks, the barangay captain or his council members accompany the prospectors to the suspected area and samples are gathered according to the basic knowledge on clay pros pecting. The promise to inform them of the results and accompanying finish products become a gentleman's agreement.

Samples are gathered by digging such that the overburden is removed. Sacks are properly labeled and the moisture of the clay is determined upon arival and the residue test is determined before any other testing follows. It takes at least two days to gather samples in one barangay depending on the guide and the people in the locality. Some of the barangay captains are reluc tant to cooperate in the survey for some reasons, but most of them are cooperative, believing that good things will happen in their barangay in the near future.

#### 2. Material Source/Preparation

Clay minerals used in the study come from Iligan City, Lanao del Norte, Cagayan de Oro City and Misamis Oriental, Butuan City, Agusan del Norte and Misamis Occidental. The color of the raw clay varies from brown, black, an

All the samples are packed in cement bags containing approximately 30 kilograms, enough for a person to carry from the sampling site. The residue test was conducted on dried samples using the 200mesh (74 micron) screen. The screen oversize was dried in an oven at 110<sup>o</sup>C and weighed. The screen oversize will serve as the basis, whether the clay will be formulated prior to forming or will be used directly as single or monobody clay.

#### 3. Shaping Molding

The test pieces were prepared using the soft mud method with 25-35% water of plasticity. The added water was measured using a graduated cylin der before the forming process and after the forming process using the mois ture analyzer. Kneaded samples stay to remain overnight before the forming of test specimens in order to achieve the uniform moisture content of the clay. Clay samples were shaped in a 22 x 6x 1.5 cm metal mold for modulus of rupture test (MOR) and in 2.5 x 2.5 x 4 cm metal mold for the compres sive test. A 5 cm marked and 3 cm mark were measured on the sample for MOR and compressive tests, respectively. Five samples were made for every characterization conducted.

#### 4. Drying/Firing

Test pieces were dried at room temperature for one week and in an Test pieces were dried at room temperature for one week the<br>ven at 110<sup>o</sup>C overnight before firing in order to completely remove the mechanically held moisture. Changes in the dimensions were measured after drying and after firing to determine the drying and the firing shrinkages respectively. Dried samples were fired in the "Thermolyne" electric kiln at 750C, 850°C, 950°C, 1050 °C and 1150°C.

#### Testing Evaluation Š.

1. The residue of the clays such as mica or sand that is associated in<br>the clay was determined by passing the slaked clay in a 74micron<br>(200mesh) screen. The residue was dried and weighed. Percent resi-<br>due was calculated

$$
\% \text{ Residue on 74micron (200 mesh)} = \frac{\text{wt. of residue}}{\text{wt. of clay samples}} \times 100
$$

2. Water of plasticity was determined after the clay was kneaded and the workability of the clay was achieved. A clay sample of 2.5 x 2.5 X 4 cm was molded and dried overnight in an oven at 110 °C. The cubes are weighed as soon as they are cooled and the percent water of plasticity was calculated using the formula as follows. A direct method of determining the water of plasticity is by using the mois ture analyzer.

% H,0 of Plasticity wet weight - dry weight dry weight -x 100

3. The final drying of the test pieces was at 110 °C overnight in an oven. The linear drying shrinkage as a percentage of dry length was calculated as follows:

$$
S_{d} = \frac{L_{i} - L_{d}}{L_{i}} \times 100
$$

Where:

 $S_d$  = linear drying shrinkage in percent

 $L_1$  = initial length of test piece<br> $L_d$  = dry length of test piece

measured using the "Shimadzu Universal Testing Machine and <sup>4</sup>. The dry modulus of rupture or the breaking strength (MOR) was was calculated as follows:

Breaking Strength 3 PL  $2 wt<sup>2</sup>$ 

#### Where:

- $P =$  maximum load at breakage of the test piece
- $L =$  distance between lower supporting points
- $w =$  width of test pieces
- $t =$  thickness of test piece
- 5. The firing shrinkage and the total shrinkage were measured as fol lows:
- 4. Firing Shrinkage

$$
S_d = \frac{L_d - L_f}{L_d} \times 100
$$

Where:

 $S_i$  = linear shrinkage after firing

 $L_d$  = dry length of test piece

- $L_f$  = fired length of test piece
- B. Total Shrinkage

$$
S_t = \frac{L_t - L_t}{L_t} \times 100
$$

Where:

- $S<sub>t</sub>$  = total linear shrinkage after drying and firing
- $L_1$  = initial length of the test piece  $L_f$  = fired length of the test piece
- 
- UTM and was calculated as follows. 6. Compressive strength was also performed using the "Shimadzu"



Where:

- $P =$  maximum load at breakage of the test piece  $A = cross sectional area$
- 7. Water of absorption test was conducted by boiling the test pieces. The samples were completely immersed in water but not touching the bottom of the container. The samples were cooled for 12 hours while completely immersed in water. The samples are wiped off lightly of the water drops and weighed. The percent water absorption was determined as follows.

% Water Absorption = 
$$
\frac{W_2 - W_1}{W_1} \times 100
$$

Where:

 $W<sub>2</sub>$  = wet weight of the test pieces in grams  $W_i$  = dry weight of the test pieces in grams

Visual inspection was made on every sample to detect the presence of hairline cracks or any defects arising from or inherent from the location of clay.

#### Results and Discussion

The results reveal that there are clays suited only to certain product applications when used as a single body, but others can be made into ceramic items and meet the necessary standards of the product. Generally. all the clay samples that meet the minimum requirements or the sand residue can be a Source of raw materials in the manufacture of pots, jars and othe: terra cotta items.

The result of the residue test is presented in table l. The amount of residue ranges from 5.75 to 41.46 for samples LLA and LJA respectively.

Two samples, namely LLA (5.75), and DLN (10.68) have residues less than<br>20 weight percent, with brown color and brittle texture. Two samples con-<br>taining residues within the 20% range, as follows BRY (20.13) and LGT<br>(21.1 samples contain residues within the 40%, namely LA1 (37.21) and LJA  $41.16$ ). In the preliminary studies conducted on these clays, namely LLA and DLN. we observe that the appearance of hairline cracks or warping (Ibarra on press) is due to the insufficient amount of sand component present in the clay. In order to solve the problem, these clay samples are formulated with screened river sand passing the 20 mesh (883micron) screen in the ratio of 70:30 clay and sand, respectively.

No.	Code	Location Retained %	Weight Texture	Residue Color/
	<b>LLA</b>	Lanao del Norte	5.75	brown, brittle
$\overline{c}$	<b>DLN</b>	<b>Butuan City</b>	10.86	brown, brittle
3	<b>BRY</b>	Lanao del Norte	20.13	white to brown, strong
$\overline{4}$	LGT	<b>Misamis Oriental</b>	21.17	brown, strong
5	LGN	Misamis Oriental	28.28	brown, strong
6	LA2	<b>Butuan City</b>	29.09	white to brown, strong
7	<b>DLG</b>	<b>Butuan City</b>	29.17	black, strong
8	<b>BLA</b>	Cagayan de Oro City	30.40	brown, strong
9	<b>PGN</b>	Iligan City	31.28	brown, strong
10	LA1	<b>Butuan City</b>	37.21	white to brown, strong
11	LJA	Misamis Occidental	41.46	white to brown, strong

Table I. Weight Percent of Clay Retained on 200mesh (74 microns)

Table 2 presents the particle size distribution (cumulative percent retained) of the 200mesh screen oversize for all the clay samples except for samples LLA and DLN. The data are sorted in ascending order in the 150mesh amples LLA and DLN. The data are sorted in ascending order in the respectively.<br>
column. The sorted data show that samples BLA  $(81.91\% \text{ retained})$  and LGN (94.35%) contain the coarsest and the finest sand residue, respectively.



# TABLE 2. Cumulative Pevcent Retaincd

The property of the clay is often correlated with the particle size dis tribution of the accessory minerals, such as quartz, mica and feldspar, which are found in this particle size distribution (Salmang, 1964).

Figure I plots the relationship between the mesh number and the percent cumulative retained. All the samples, except BLA, differ only slightly in the particle size distribution.



Figure 1. Piot of Percent Cumulative Retained vs. Mesh Number

It can be clearly observed that, the curve of sample BLA is distinct and separated from the curves of the remaining samples. Except for sample BLA the curves of the remaining samples representing the percent retained in the 28-150mesh, vary slightly.





52

The water of plasticity ranges from 21.40 to 35.23%. Both samples<br>of LLA and DLN were mixed with river sand resulting to a reduction of of LLA and tempering water by approximately 8%. Samples LGN and LGT contains 28.28 and 21.17% residue respectively. Sample LGN has water of plasticity of 33.20% and a drying shrinkage of 12.47% while sample LGT has a water of plasticity of 35.23 and a drying shrinkage of 16.70%. The amount of residue of sample LGT is comparable to sample BRY, however, the water of plasticitv is about 10% higher than sample BRY. Moreover, these two samples have almost similar amount of cumulative percent retained of 91.72 and 92.36%. respectively. The reason for this observation is related to the location of the source of clay. The mountain where this clay is located is the source of an amorphous silica used in the production of cement and the nearby mountain contains limestone minerals. On the other hand, sample LGN and LSA I have comparable amount of residue but differ by about 7% of tempering water. This observation can be traced to the mountain where the clay is located, which contains limestone minerals. Therefore, LGN and LGT, which are town apart and where the cement plants are located will have similar properties.

Table 4 tabulates the modulus of rupture (MOR) of the clay samples. Out of the 11, samples only 9 samples are tested for MOR. Samples LGN and LGT exhibit hairline cracks in all firing temperatures. Although the sand residue is within the range of good clay (Ibarra, unpublished), the high water of plasticity indicates the possibility of cracking on the surfaces during fir ng. Ihe most probable reason is the presence of limestone in the clay, where calcination takes place at higher temperatures. Upon visual inspection on the crack surfaces, white spots or blisters are clearly observed; thus, it supports the probability of the presence of limestone. The same phenomenon is observed to clay samplesgathered from the towns of Albur and Sagbayan in the province of Bohol (Ibarra, unpublished). Samples DLN and LLA exhibit the two lowest values of MOR, with highest value within the  $20 \text{ kg/m}$ em<sup>2</sup>. These are clays added with river sand, however, the amount of water of plasticity and the drying shrinkage are relatively high, with values that are more than 12 percent. Even with the present data, samples LLA and DLN can already be utilized in the production of pots and bricks. The high value<br>of linear detailed in the production of pots and bricks. The high value sand is to be added. Sample LLA results show that it needs the addition of<br>non-plastics non-plastics, specifically fluxes. Generally, the low MOR data is possibly of linear drying shrinkage of sample DLN is an indication that more river<br>sand is to be

No.	Code	Modulus of Rupture, kg/cm <sup>2</sup>					
		750 <sup>°</sup> C	850°C	950 °C	1050 °C	$1150\degree C$	
2	DLN	18.06	19.84	12.42	10.72	17.42	
	<b>LLA</b>	19.50	19.93	19.56	15.84	21.05	
6	LA2	27.55	21.35	23.16	20.15	30.48	
7	<b>DLG</b>	28.19	28.89	27.69	23.55	34.38	
$\Omega$	PGN	30.12	29.42	30.85	35.72	36.37	
10	LAI	34.77	35.46	32.92	47.32	50.64	
3	BRY	38.87	38.12	36.34	48.96	73.40	
11	LJA	46.63	44.82	42.36	53.82	92.45	
8	<b>BLA</b>	50.91	45.71	54.44	58.46	110.23	

Table 4. Modulus of Rupture of Clay Samples

affected by the fact, that the samples are first subjected to water absorption test before MOR.

Figure 2 shows the plot of MOR data. Two observations are deduced from the graph. First, there is a point in the figure that shows a decrease in MOR between 900 to 1000°C, second, above 1050°C, the curves generally



Figure 2. Graph on the Modulus of Rupture

increase upward. This observation is due to the changes in clay substance within the temperature range, especially in the impurities present in the acessory minerals such as, the polymorphic transformation of silica (Salmang 1961). Investigations regarding this phenomenon can be conducted by using grog in place of river sand. The general increase in the MOR above 1000  $^{\circ}$ C may be due to the progress in the densification, as indicated by the increase in firing shrinkage and reduction in the water absorption.

The compressive strength of clay samples is summarized in table 9. Sample LLA, the clay that contains the lowest amount of sand residue, also yielded the lowest value in compressive strength, even with the addition

No.	Code	Compressive Strength (kg/cm <sup>2</sup> )					
		750 °C	850 °C	950 °C	1050 °C	1150 °C	
	<b>LLA</b>	45.84	58.37	64.47	65.10	66.97	
$\overline{2}$	<b>DLN</b>	123.23	122.33	122.92	83.86	83.60	
8	<b>BLA</b>	116.92	135.79	128.00	70.64	116.02	
6	LA2	92.06	104.12	99.46	66.69	131.44	
9	PGN	122.76	132.28	135.40	120.34	147.24	
10	LA <sub>1</sub>	110.23	115.24	120.13	95.64	154.22	
3	<b>BRY</b>	113.80	125.42	140.78	162.78	166.08	
11	LJA	139.90	160.50	156.42	137.12	266.32	
	<b>DLG</b>	140.94	242.83	251.40	298.98	347.12	

Table 5. Compressive Strength of Clay Samples

of river sand. The value can be possibly remedied by adding more of the river sand in the formulation. Compared with sample DLN, the clay also formulated with sand, resulted into a higher strength than LLA sample. Sample L

amount of residue, with 41.46% and with 93.14% retained in 150 mesh.<br>DLC  $\frac{150 \text{ m}}{150 \text{ m}}$ DLG, unexpectedly, resulted with the highest strength, rising continuously in<br>all the firing temperatures. This is the only sample that showed the type of<br>behavior similar to sample D (Navarro et. al. 1983) and sample no.1

Vol.  $XV$ . No. 1

et. al. 1988). The observations in sample DLG agree with sample  $D$  of  $L_{u_{z_0}}$  clay, i.e., both have the highest drying and firing shrinkage. It also contains clay, i.e., both have the high-<br>the high-amount of clay minerals. Another possible factor that needs to be<br>the kind of  $\alpha$ the high amount of clay minerals. Another possible factor that needs to be investigated that contributed to its high strength is the kind of sand residue, the only sample that contains black residue.<br>Similar observation w

data in relation to firing temperature, i. e. there was a decreased of both strengths between  $900$  to  $1000^{\circ}$  and again the strength decreased of both strengths between 900 to 1000°C and again the strength increased above<br>1050°C. As mentioned earlier, the effect may be attributed to the presence of<br>non-plastics especially quartz mineral, that transform into tridymite, r Similar observation was found to the MOR and compressive strength





The increase in weight, expressed as percentage of dry weight or water absorption is presented in table 10. Sample LLA yielded the highest water<br>absorption volume (10.000). Sample LLA yielded the highest water lewest values in MOR (17.42 kg/cm<sup>2</sup> at 1150°C) and compressive strength (66.97 kg/cm<sup>2</sup> at 1150°C) and compressive strength absorption values (18.07% at 1150°C) for all the firing temperatures and had<br>lewest values in MOS.  $(66.97 \text{ kg/cm}^2 \text{ at } 1150^{\circ}\text{C})$ . It means that there are no s substantial changes

curring inside the structure of the body, therefore, the clay contains insufficuring mount of fluxes to form the glassy phase at higher temperatures. The degree of shrinkage and porosity of sintering clays depend on the amount fluxes present and on the ratio of  $Al_2O_3$  to  $SiO_2$  (Salmang, 1961).



# Table 6. Water Absorption of Clay Samples

The lowest water absorption is observed in sample LA 2 with a value of 6.95% at 1150°C, with a corresponding compressive strength of 131.44 kg/cm<sup>2</sup>. The sample reaches its vitrification temperature, as evident by the texture and the chocolate brown color. The body can possibly be formulated with little addition of fluxes to be used as a raw material for red clay stoneware body. Ovenware, red stoneware and other stoneware products With a well-integrated body-glazed interface may be classed as "stoneware" even if the porosity is as high as 10% (Ryan et. al. 1987). The percent reduction of water absorption ranges from 5.04% to 65.61% at 1150°C. Clays LAI, LA2, and DLG, with minimal addition of non-plastics are potential red clay bodies for stoneware products. Water absorption and firing temperature are plotted in figure 4. Generally, it can be observed that water absorption decreases with increasing firing temperature. Taking a closer observation in the different temperature increment, the water take-up of the all clay samples except DLN and LSA2, increases up to as high as 13% for sample from 750-850 °C. Half the number of the samples still increase their water take-up within the

Finally, above 1050°C all clay specimens exhibit the reduction in the water<br>absorption.<br>The clays show different patterns of water absorption. Some of the 850-950°C intervals. On the other hand, samples BLA, BRY, PGN, DLG and LA2 start to decrease their water absorption. Within the 950-1050°C inter-LAZ start to decrease their water absorption

clays continue to increase their water take-up while other clays are in the stages of reduction. Sample BRY continues to increase its water take-up until 1050°C. This indicates that the clay is refractory or it contains l stages of reduction. Sample BRY continues to increase its water take-up unmaterials. The body that easily vitrifies is sample LA2, where the water absorption curve starts to decrease even at 850°C. The total decrease of the water of absorption is 65.51% from 750 to 1150°C. This will probably indicate the high amount of fluxing agent present in the clay in contrast to sample BRY. The difference in the percent decrease of the water of absorption is also an indication of the amount of the fluxing inherently present in



Figure 4. Water Absorption Plot of Clay Bodies

58

the clay deposits, which has an important role at higher temperature. The diminution of water of absorption appears to be the characteristic of all ceramic clays. It signals the start of the vitrification process.

### Conclusion

Clay mineral prospecting using the basic test on residue determina tion by screening into 200 mesh (74 microns) proves to be one of the effective tools in testing the red clay for pottery and brick production. The amount of sand retained in the 200mesh screen, which is from 20 to 45%, shows very satisfactory results in the physical and mechanical characterization tests. The amount of tempering water for potential raw material red clay body ranges from 20 to 30%. Red clay drying shrinkage for commercial application is observed within the 10% limit. Majority of the clays tested in compressive strength qualify in the second grade of ASTM standards, such as clays BLA, PGN, BRY, LAl and LJA. Clay DLG's mechanical property is within the first grade of ASTM standard and clay LA2 for the third class classification. The modulus of rupture of the samples is highest in the clay that contains higher amounts of coarser materials as in sample BLA, where it contains the highest amount of cumulative percent retained in 150mesh (100 microns).

# Recommendations

Based on the results of tests performed, the proposed tasks to be con ducted in the future are the following:

- I. bench scale testing and production using the actual conditions in the production prior to commercialization;
- 2. formulation studies of potential clays for higher end products such as stoneware and roofing tiles; stoneware and roofing tiles;
- 3. conduct extension services for clay mineral prospecting to determine clay deposits for utilization in ceramic livelihood projects;
- +. utilization of fluxing agent present in the clay in contrast to sample BRY. The difference in the percent decrease of the water of absorp tion is also an indication of the amount of the fluxing inherently present in the clay deposits, which has an important role at higher tempera ture. The diminution of water of absorption appears to be the charac

teristic of all ceramic clays. It signals the start of the vitrification process "fat" clays by blending with "lean" clays or by adding fillers from waste products.

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