

IMPROVING TAPIN KAOLIN QUALITY FOR WHITE WARE CERAMIC

PENINGKATAN KUALITAS KAOLIN TAPIN UNTUK BARANG KERAMIK PUTIH

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ABSTRACT

Tapin kaolin cannot directly be utilized as raw material for white ware ceramics due to its high Fe₂O₃ content. It needs upgrading its quality in terms of fulfilling the specification of white ware ceramics. Such the material requires a series of process including washing, wet sieving in magnetic ferro-filter equipment and then dissolving by H₂SO₄ 10%. Based on several parameters such as Al₂O₃, SiO₂ and Fe₂O₃ contents, 2- μ m particle size density, whiteness and plasticity; Tapin kaolin quality develops significantly. XRD analysis shows that the material does not have maghemite anymore. Quartz is relatively low and mafic minerals are unavailable. Referring to such quality, Tapin kaolin can be used for Parian porcelain.

Keywords: Tapin kaolin, upgrading, white ware ceramics, and Parian porcelain

SARI

Kaolin Tapin, Kalimantan Selatan tidak bisa langsung dimanfaatkan sebagai bahan baku untuk produk keramik putih, karena kaolin masih tinggi kandungan Fe₂O₃-nya. Agar memenuhi spesifikasi sebagai bahan baku keramik putih, material ini perlu ditingkatkan kualitasnya dengan cara pengolahan bertahap mulai dari pencucian, pengayakan basah menggunakan alat magnetic ferro filter dan dilanjutkan dengan pelarutan menggunakan H₂SO₄ konsentrasi 10 %. Hasil proses menunjukkan kualitas kaolin yang meningkat dilihat dari kadar Al₂O₃, ukuran partikel pada 2 μ m, densitas, derajat putih, dan keplastisan serta kandungan SiO₂ dan Fe₂O₃ berkurang. Kaolin terolah diuji dengan analisis XRD. Hasil uji menunjukkan tidak dijumpai adanya mineral besi maghemite. Uji SEM menggambarkan fasa dominan adalah mineral kaolinit sedangkan kandungan kuarsa relatif rendah; tidak terdapat mineral gelap. Mengacu kepada hasil uji, kaolin proses ini dapat digunakan sebagai bahan baku keramik putih jenis bodi parian seperti keramik hias dan alat makan minum (tableware).

Kata kunci : kaolin Tapin, proses pengolahan, keramik putih, porselen parian

INTRODUCTION

Kaolin is one of the main raw materials besides feldspar, quartz and ball clay for ceramic making. SNI 06 0578 89 specifies that kaolin for fine ceramic should have Fe₂O₃ = 0.4 – 0.8%, TiO₂ = 0.3 – 0.7%, SO₃ = 0.2 – 0.4% and white-

ness (minimum) 80 (Anonymous, 1989). Such a specification, categorized as high quality kaolin, is available at Bangka-Belitung and has commercially been utilized for ceramic industry in Java and other places as ceramic binder and filler. Yet

reserve of such a deposit gradually decreases. As a result, it needs seeking new kaolin deposit. One of the promising deposits is available at South Kalimantan.

Tapin Regency at South Kalimantan possesses 27.2 million tons kaolin deposit that is available at Bitahan and Binderang while 21.3 million tons occurs at Lokpaikat districts and 5.9 million tons at Tatakan, South of Tapin district [Anonymous, 2005a]. However, kaolin from the mentioned areas performs poor quality and does not satisfy the requirement for white ware ceramics. White ceramics requires Fe₂O₃ and clay mineral contents around 0.94 – 3.87% and 43.15 % respectively (Anonymous, 2005b). If the kaolin comprises impurities such as quartz, mica, iron oxide and organic matter, it cannot be used for ceramics, notably white ware body.

In order to improve the quality of Tapin, beneficiation is required. The process includes physical magnetic (elutriation and sedimentation methods) as well as chemical treatments. Elutriation and sedimentation processes are based on Stokes's law. Particles with lesser size but bigger density will settle fast than that of bigger size but smaller density. Kaolin processing at Belitung Utama Company in Bangka-Belitung applies elutriation process. Subari et al (2009) states that physical method for kaolin improvement is started by preparing the slurry followed by sieving process to obtain size from 0.5 mm up to 325 meshes. The Fe₂O₃ content is minimized by magnetic ferro filter (Tahli, 2008). If the ferro magnetic instrument is not available, the process can be conducted by dissolving the material in H₂SO₄ or HCl 10% solution.

Magnetic concentration is based on the difference of magnetic character. Natural mineral owns three magnetic characters (Wills, 2006; Nuryanto et al., 2003). Those are strong magnetic (magnetite, hematite, franklinite and ilmenite), weak magnetic (siderite, pyrite, limonite and marcasite) and non magnetic (quartz, feldspar, dolomite and barite).

Grouping minerals by its magnetic intensity belongs into two types, namely low and high magnetic intensities. The former is normally used for purifying material through dry method while the later is applied for separating material through wet method. The magnetic ferro filter or MFF is one of tool types using high magnetic intensity.

Based on mineral composition, low grade kaolin contains kaolinite 31.1%, quartz 31.05%, potassium feldspar 29.6%, montmorillonite 3.8%, illmenite 1.1% and other minerals 2.8% while high grade kaolin comprises of kaolinites 68.9%, quartz 4.1%, potassium feldspar 15.3%, montmorillonite 7.6%, illmenite 1.3% and other minerals 2.8%. Chemical compositions of both kaolins are shown in Table 1 (Hojamberdiev et al., 2005).

The objective of this research is to improve the quality of Tapin kaolin to be used as raw material for white ware ceramics of Parian body as well as to prepare raw material for Ceramic industries at South Kalimantan and another area. Parian - from the Greek island of Paros - is a white ware ceramics body that has self glazing property that gives marble appearance. Its composition consists of kaolin, feldspar and ball clay (Arthur, 1973).

In case the research is promising, processing technology of kaolin quality improvement can be scaled up into pilot plant level by involving local government and or interest investor and collaborating with Bandung Center for Ceramics.

METHODOLOGY

Quality improvement and beneficiation processes of Tapin Kaolin include several steps. Those are:

1. Preparing natural kaolin as raw material. Such preparation comprises of drying and quartering. Drying process is conducted to reduce the moisture content within samples. Around 125 kg kaolin is dried in an oven or direct sunlight. When dried, the samples size

Table 1. Chemical composition of low and high quality kaolin

Kaolin Type	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	Na ₂ O	K ₂ O	LOI
	%								
Low quality (unprocessed kaolin)	70.5	17.5	0.73	0.45	0.73	0.15	0.31	3.7	5.75
High quality (processed kaolin)	51.0	33.8	0.47	0.34	0.48	0.49	0.19	1.48	11.70

is then reduced into less than 2 mm. The next step is quartering the samples to make them homogenous. To quarter a sample, first mix and pile the sample on a canvas, using a shovel. Place each new shovelful on the top-center of the preceding one so that the soil will be distributed evenly in all directions. Then flatten the sample to a circular layer of approximately uniform thickness. Next, insert a stick or length of pipe under the canvas and then lift it at both ends to divide the sample into two equal parts. Remove the stick, leaving a fold in the canvas, and then reinsert it under the sample, but this time, at right angles to the first division. Again, lift the stick. This divides the sample into four parts. Discard two diagonally opposite quarters, taking care to clean the fines from the canvas. Then remix the remaining material, taking alternate shovelful from each quarter. Repeat the quartering process as necessary to reduce the sample to the desired size. This research requires 60 kg and 50 kg with 2.5% water content. The 60-kg sample then goes to testing laboratory for physical characteristics tests and beneficiation process while the 50-kg sample is kept as an archive to anticipate sudden need of raw material when the experiment for ceramic making is unsuccessful;

2. Improving kaolin quality. Figure 1 illustrates beneficiation steps for Tapin kaolin;
3. Characterizing raw and processed kaolin. Characters to be tested include fired test at 1.400°C, whiteness, density, plasticity, particle analysis by Andreasen method, dry shrinkage and bending strengths, chemical , mineral and SEM analyses;
4. Designing parian body composition. Several materials can be used to make up parian body such as enriched kaolin, feldspar and ball clay. Table 2 exemplifies composition design

- of parian bodies as stated by Arthur (1973);
5. Formulating test piece. Specimens around 10 cm x 10 cm are made using designed formula as stated in Table 2 and hydraulic press at 200 kg/cm². Temperature firing within kiln varies. It depends on its designed formula. As the specimens are made for laboratory scale, the employed kiln may be electric or gas kiln.
6. Characterizing specimens. Properties to be tested for this specimens are shrinkage, density, porosity, water absorption, hardness (Moh's scale), sound, fired color, chemical and mineral analyses and SEM/EDX tests;
7. Making parian body prototype. The prototype is made by burning Tapin kaolin at 1.150 – 1250°C. Its process is as follows:
 - preparing raw materials, i.e., feldspar, ball clay, and enriched kaolin;
 - weighing each raw material based on designed formula;
 - mixing the material by adding water around 5 – 7%;
 - casting the art ceramics and table ware using gypsum mold;
 - drying the products in the oven;
 - firing the products at a given temperature.

RESULTS AND DISCUSSION

The quality of Tapin kaolin is relatively low. Therefore its quality needs to be improved.

Procedure to improve the quality of Tapin kaolin is shown in Figure 1. Experimental data obtained from the tests for original kaolin is shown in Table 3. The data show that quartz is the dominant mineral as impurity material available in sieve while from 252 g sample analyzed by the MFF state that Fe₂O₃ is the dominant impurity.

Table 2. Designed formula for parian body

Model	Raw materials (%)		
	K-feldspar	Pure Tapin kaolin	Tapin ball clay
1	40	50	10
2	45	45	10
3	50	40	10
4	55	35	10
5	60	30	10

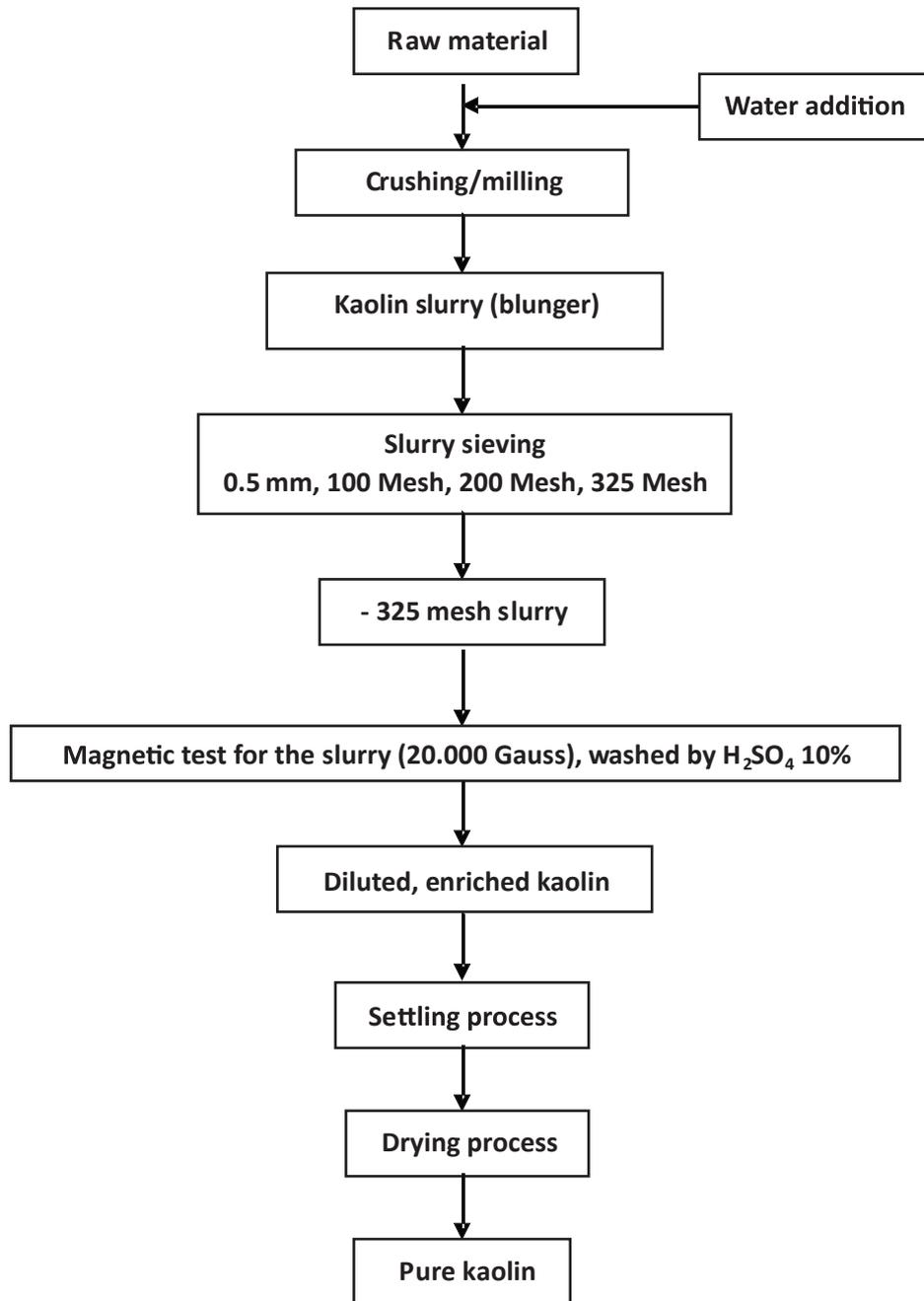


Figure 1. Flow charts for beneficiating Tapin kaolin

Tapin kaolin is different from Belitung one. Of 10 kg Tapin kaolin, around 1.386 kg belongs to impurities (quartz sand, iron oxide and organic matter), 8.410 kg goes to clean kaolin and the rest is significant lost during the process. Meanwhile, of 20 kg Belitung kaolin, 9.20 kg goes to impurities, 10.48 kg serves as clean kaolin and the rest is vanished during the process.

Magnetic test using MFF (Figure 2) is conducted at laboratory scale to reduce the iron oxide. The employed magnetic field is 20,000 gauss. Procedure for magnetic test is shown in Figure 1 and the result is illustrated in Table 3. The average material in MFF is 252 g.

Table 3. Experimental data of Tapin kaolin beneficiation

Experiment no.	Kaolin weight (kg)	Water volume (liter)	Impurities in sieve (g)				Materials in MFF Weight (g)
			0.5 mm	100 mesh	150 mesh	325 mesh	
1	10	20	125	114	27	738	400
2	10	20	104	111	32	605	170
3	10	20	108	53	95	987	250
4	10	20	110	112	60	776	310
5	10	20	123	120	56	1214	120
Average Number			114	102	54	864	252



Figure 2. Magnetic ferro filter instrument

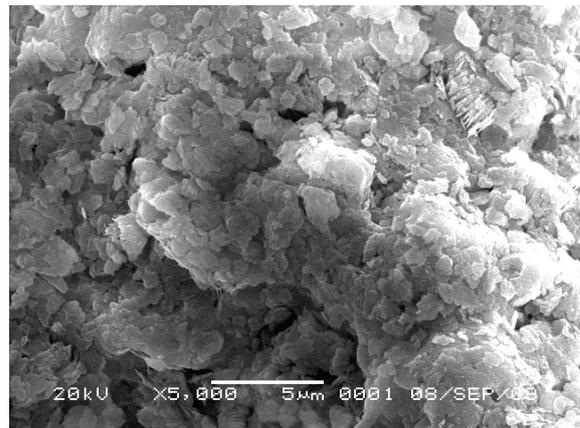


Figure 3. Photomicrograph of unprocessed kaolin

Figure 3 and 4 show the SEM analyses for raw and processed Tapin kaolin respectively. Compared to quartz (Figure 3), the existence of kaolinite within detected sample is less than that of quartz. The material is available at upper right side (red circle). On the contrary, kaolinite seems abundant within the processed Tapin kaolin. It is characterized by plated character (blue ellipse). Table 4 represents result of EDX tests for raw and processed kaolin. It is clear that the Al_2O_3 content within the processed kaolin is bigger (31.04%) compared to similar material within raw kaolin (19.70%). Not only SiO_2 content decreases (62.08%) within processed kaolin but also FeO content reduces (0.27%). Washing and sieving seem responsible for increasing Al_2O_3 content and decreasing SiO_2 matter. Of 10 kg raw kaolin, 1.134 kg belongs to quartz impurity AAS analysis for Tapin processed kaolin show the increase of Al_2O_3 content up 11.03% but the SiO_2 and Fe_2O_3 contents decrease to 11.69% and 0.98% respectively (Table 5). the result of EDX and AAS analysis of raw kaolin and processed (enriched) kaolin are significantly similar.

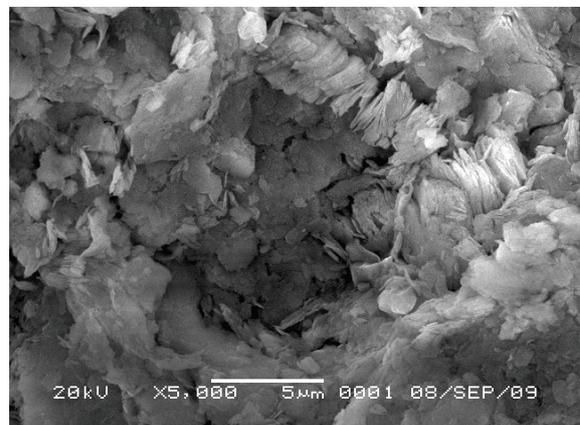


Figure 4. Photomicrograph of processed kaolin

XRD analyses conducted to Tapin kaolin for both raw and processed ones. Figure 5a and b illustrate XRD diffractogram for both materials. Alpha quartz, kaolinite, feldspar and maghemite are minerals available within raw material or unprocessed kaolin (Figure 5a). Feldspar and maghemite are unavailable in clean or processed kaolin (Figure

Table 4. EDX analysis results for Tapin kaolin

Material	Composition (%)			
	Al ₂ O ₃	SiO ₂	FeO	C
Unprocessed kaolin	19.70	71.92	1.30	0.08
Processed kaolin	31.04	62.08	0.27	3.61

Table 5. AAS analysis results for Tapin kaolin

Material	Composition (%)								
	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	TiO ₂	CaO	MgO	K ₂ O	Na ₂ O	LOI
Unprocessed kaolin	19.71	68.98	1.29	1.13	0.34	0.22	0.48	0.09	4.46
Processed kaolin	30.97	58.29	0.31	1.00	0.19	0.12	0.20	0.16	10.25

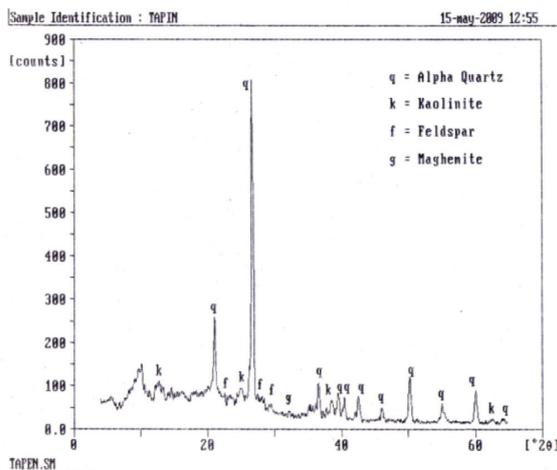
5b). The dominant quartz in kaolin (Figure 5a) will cause low shrinkage property in ceramic body during sintering at above 1100°C. On the contrary, pure kaolin with low Fe₂O₃ (0.72 %) is suitable for white ware ceramics when combined with plagioclase feldspar and ball clay (Ibrahim et al., 2006).

Table 6 shows quantity of each minerals for either raw (unprocessed) or clean (processed) Tapin Kaolin. It is clear that SiO₂, Fe₂O₃ and TiO₂ decrease when the Tapin kaolin is diluted in H₂SO₄ 10%. Not only does the quantity of three components decrease but also the content of Al₂O₃ significantly reduces. Kaolinite increases from 34.39% in unprocessed kaolin to 71.56% in processed one but quartz decreases around 26.01%. No

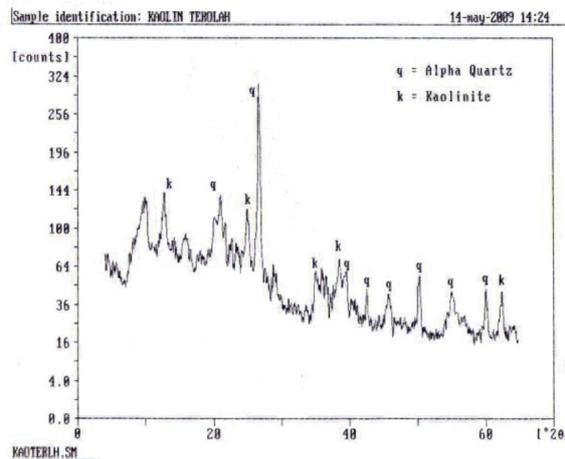
Table 6. Mineral compositions of Tapin kaolin

Analyzed samples	Available mineral	%weight
Unprocessed kaolin	Kaolinite	34.39
	Alpha Quartz	54.45
	Feldspar	10.01
	Maghemite	1.15
Processed kaolin	Kaolinite	71.56
	Alpha Quartz	28.44

iron mineral occurs within the processed material. This means that such a material satisfies for white ware ceramics making.



a



b

Figure 5. Diffractogram graph of unprocessed kaolin (a) and processed one (b)

Sumer (1999) states that generally kaolin impurities include TiO_2 , Fe_2O_3 , Na_2O and K_2O . If ceramic body contains Al_2O_3 and SiO_2 , it will then be contaminated by TiO_2 and Fe_2O_3 . As a result, the ceramic cannot be white as desired when fired. The oxide tends to be mullitization phenomenon. Agrawal et al. (2008) studies the bound of Fe_2O_3 solid solution available in mullite synthesis of 5.7% weights, temperature of $1.300^\circ C$ and tries to break it as the Fe^{3+} can replace the Al^{3+} due to its similarity in content and size.

When fired at $1400^\circ C$, original kaolin performs creamy and black spots while the processed kaolin provides milky white. No black spots occur. Compared to Bangka-Belitung kaolin that is fired at same temperature with Tapin kaolin, the result is similar. Specimens is made from processed Tapin kaolin. Its dimension is $1 \times 2 \times 10$ cm and then fired at $1,300^\circ C$. Modulus of rupture test for such specimens is $73.54 - 82.15$ kg/cm². Widjaja et al. (1996) who studied ceramic raw materials on the Island of Belitung, Indonesia found that the modulus of rupture test for Belitung kaolin is $76.12 - 91.80$ kg/cm². Referring to such data, Belitung kaolin seems more solid compared to processed Tapin kaolin. High alkali and earth alkali contents within Belitung kaolin are supposed to be responsible for this condition.

Table 7 shows whiteness tests for raw and processed kaolins. Tapin kaolin processed by MFF and H_2SO_4 10% solution methods provides

Table 7. Whiteness data of Tapin kaolin

Analyzed samples	Whiteness
Raw kaolin	76.21
Processed kaolin:	
by washing and sieving method	79.98
by MFF method	83.62
by dissolving in H_2SO_4 10 % method	87.14

higher whiteness than that of washing and sieving method, namely washing and sieving method. Specification for white ceramics requires minimum whiteness number 80. This means that Tapin kaolin processed by MFF and H_2SO_4 10% solution methods satisfies the requirement for white ceramics.

Physical characteristics test for Tapin raw and processed kaolins include particles size, density and plasticity. Particles size analysis employs Andreasen method while density analysis is determined using analytical balance and pycnometer bottle. Kaolin plasticity is tested by Atterberg method. Table 8 illustrates the test results. The results show that unprocessed kaolin retains density around 2.59 g/cm³ while the processed on around 2.54 g/cm³. Density decrease (0.05 g/cm³) occurs due to quartz and iron contents within the processed kaolin are relatively low if compared to the unprocessed one. This fact is supported by XRD analysis at which maghemite (iron mineral) and feldspar do not exist in the processed kaolin while quartz decreases. Particle size analysis also shows that the quantity of particle with < 2 μm in size for the processed kaolin is bigger than that of the unprocessed one while the unprocessed kaolin holds plastic property (IP) lesser than that of the processed one. Based on density, particle size and plasticity characters, processed Tapin kaolin is suitable for white ware ceramic body (Hayden, 2001). Components for white ware ceramic making of Parian body also includes ball clay from Tapin and feldspar from Sukabumi, West Java. Table 9 describes composition of both materials.

Parian body is a type of white ware ceramics. Such a material utilizes processed Tapin kaolin, Sukabumi K-feldspar and Tapin ball clay as its composition. The mixture is then fired at $1,050 - 1,150^\circ C$. Of the three main components for parian body, K-feldspar seems the dominant one. Around 45-60% K-feldspar is mixed with other ingredi-

Table 8 Analyses results of particles size, density and plasticity

Material	Particles size (μm)								Density (g/cm ³)	Plasticity (IP)
	> 60	40-60	30-40	20-30	10-20	5-10	2-5	< 2		
Unprocessed kaolin	3.02	13.16	10.23	24.48	20.62	3.60	5.03	19.86	2.59	9.05
Processed kaolin	0.00	20.98	5.28	21.84	17.06	5.13	6.46	23.25	2.54	10.86

ents (kaolin, ball clay). Test of parian specimen includes bulk density, porosity, fired color and hardness properties (Table 10).

Using unprocessed kaolin as one of Parian white ceramics will result in less white appearance of the ceramics as well as black spots on Parian ceramics although the material is fired at 1,150°C (standard temperature for ceramic firing). The cause is the impurities, notably iron matter, available within unprocessed kaolin. Muller and Scherer (2001) states that using processed or pure kaolin is much better than that of employing the unprocessed or raw one. Impurity like pyrite within raw kaolin is responsible for black phenomenon

Correlation among bending strength, shrinkage and water absorption properties to firing temperature can be seen on Figure 6, 7 and 8. Figure 6 shows that the higher the firing temperature the higher the bending strength. On the contrary, the higher the firing temperature the lower shrinkage and water absorption (Figure 7 and 8). Based on Figure 9, Parian body, fired at 1,100°C, is categorized as non vitreous body since the amount of water absorption is still 7.0%. Vitreous characters is shown for specimen code 1 and 2 when the

Parian body fired at 1,150°C. The water amount for such condition is less than 3%. When the water amount improves to 3.0-7.0%, the Parian body belongs to semi vitreous character Figure 9 illustrates SEM photomicrograph of Parian body fired at 1,150°C. At 2,500 X magnification, it is shown the tridymite and cristobalite phases while mullite is not available. Increasing firing temperature to 1,300°C provides mullite and cristobalite as shown in Figure 11 for 1,300°C-fired porcelain (Agrawal et al., 2008).

Analyzing Parian ceramics using AAS shows the composition as shown in Table 11. When fired at 1,250°C, the mixture will melt. Feldspar and other components (kaolin, ball clay) unite. The water amount is still high and the bending strength is relatively low. Firing to 1,300°C results in relatively low of water amount but the bending strength is high enough.

Shrinkage characters of Parian body ceramics at high temperature is less than 10%. This implies that the composition of Parian body can be applied for art ceramic and tableware making. The shrinkage property is studied in terms of determining ceramic casting dimension made from gypsum.

Table 9. Chemical composition of ball clay and feldspar

Material	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	K ₂ O	Na ₂ O	LOI
	Quantity (%)								
Tapin ball clay	60.24	26.71	0.95	1.02	0.27	0.32	2.13	0.80	7.72
Sukabumi feldspar	72.21	15.80	0.43	0.06	0.58	0.07	6.19	3.86	1.02

Table10. Parian body characteristic, fired at 1,050-1,150°C

Specimen code	1050° C				1100° C				1150° C			
	BD	PS	FC	HM	BD	PS	FC	HM	BD	PS	FC	HM
1	1.64	15.24	white	4	1.69	10.54	white	5	2.29	4.52	white	6
2	1.69	14.85	white	4	1.70	10.12	white	5	2.23	3.13	white	6
3	1.60	14.58	white	4	1.68	11.35	white	5	2.17	6.35	white	6
4	1.59	16.21	white	3	1.65	12.74	white	4	2.14	7.46	white	5
5	1.58	16.87	white	3	1.63	13.50	white	4	2.10	8.73	white	5

where:

- BD = bulk density, g/cm³
- PS = porosity, %
- FC = fired color
- HM = hardness, Moh's scale

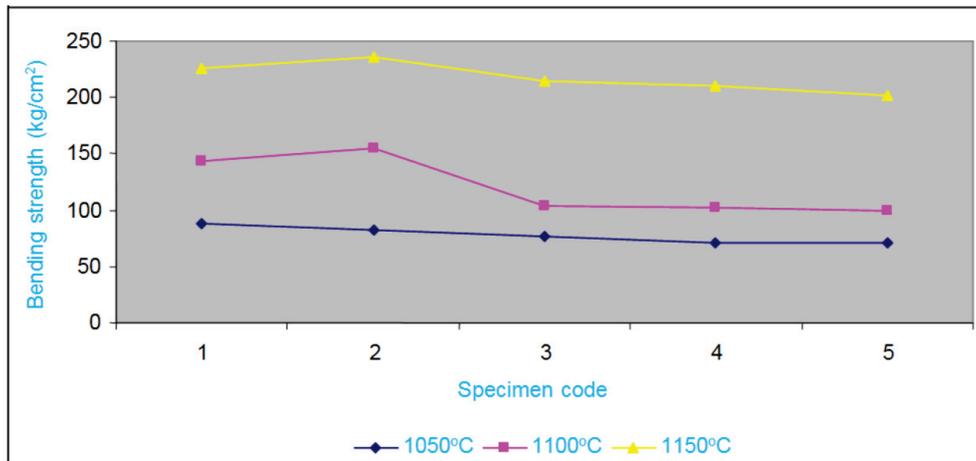


Figure 7. Correlation between bending strength and firing temperature

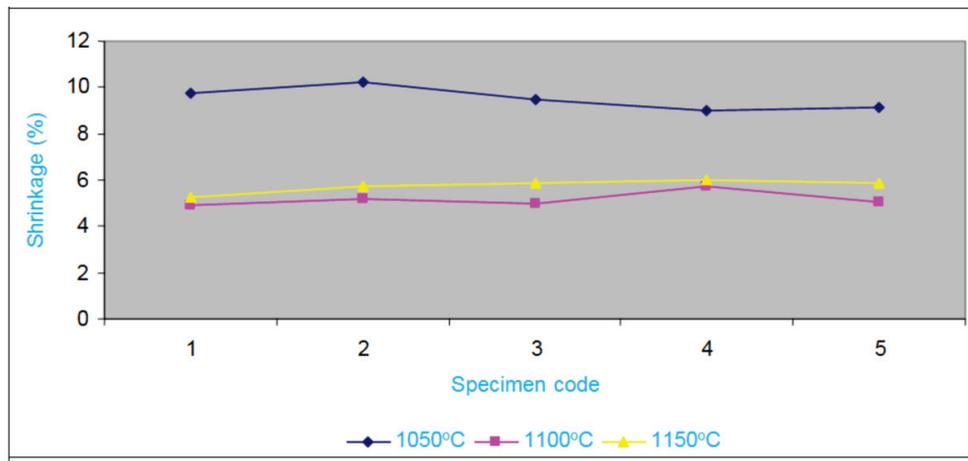


Figure 8. Correlation between shrinkage and firing temperature

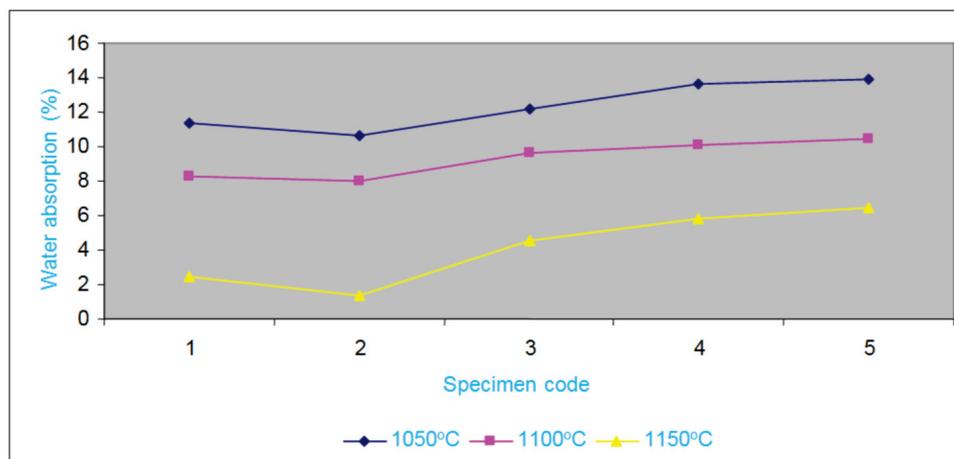


Figure 9. Correlation between water absorption and firing temperature

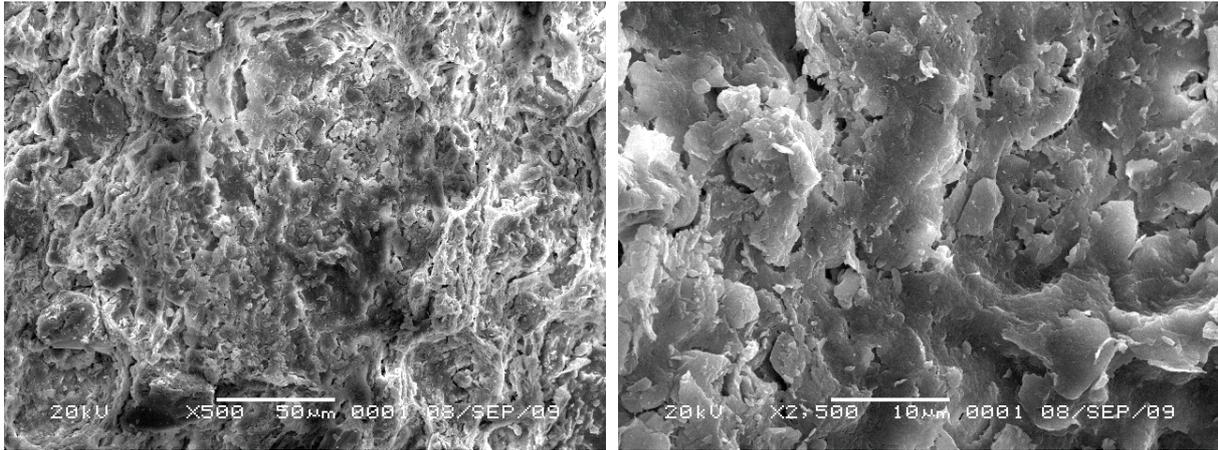


Figure 10. Photomicrograph of Parian body fired at 1,150°C

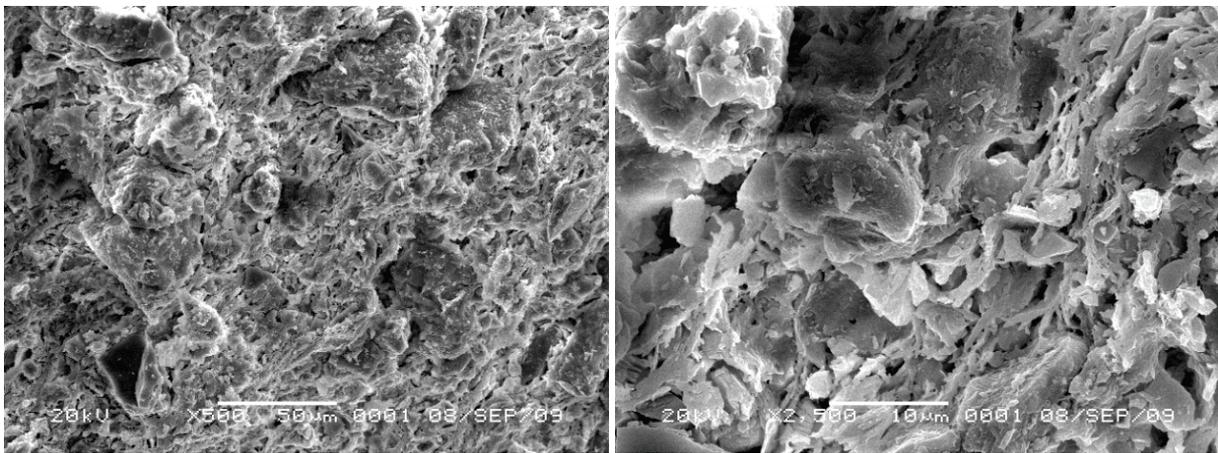


Figure 11. Photomicrograph of Parian body fired at 1,300°C

Table 11. Parian composition analyzed by AAS

Material	Composition (%)								
	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	TiO ₂	CaO	MgO	K ₂ O	Na ₂ O	LOI
Parian ceramics	16.45	72.40	0.71	1.05	0.92	0.18	0.40	0.70	0.62

CONCLUSIONS

Quality improvement occurs when Tapin kaolin is beneficiated. The process employs raw materials that retain 0.5 mm as well as 100, 150 and 325 meshes. Of the four sample types, material with -325 meshes in size is then used as representative sample and processed for iron oxide removal by MFF and H₂SO₄ 10 % dissolution. The improved quality is noted by the increase by Al₂O₃ content and the decrease of SiO₂ and Fe₂O₃ significantly as well as the increase of whiteness number.

Referring to this fact, Tapin kaolin is suitable for white ceramics.

As raw material, processed Tapin kaolin can be used for white ware ceramics either art ceramic or tableware that belong to parian body. Combination of Tapin ball clay and Sukabumi feldspar can also be utilized for white ware ceramics making. Processed Tapin kaolin, Tapin ball clay and Sukabumi feldspar are indigenous materials that can support Indonesian ceramic industries.

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