

BIODLEACHING OF AL-SOOFI AND DWAIKHLA KAOLINS FROM IRAQI WESTERN DESERT, FOR PAPER INDUSTRY

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ABSTRACT

Iraqi kaolin of Dwaikhla and Al-Soofi deposits were biobleached using the mold *Aspergillus niger*. The major discoloring of these clays is due to the presence of iron, titanium oxides and organic impurities. In this work, the Fe_2O_3 content of Al-Soofi Kaolin was lowered by 28.31% (from 2.19% down to 1.57%), while the Fe_2O_3 content of Dwaikhla Kaolin was lowered by 31.25% (from 1.61% down to 1.10%). The TiO_2 content was also lowered by 33.6% and 27.0% (from 1.16% and 1.48% to 0.77% and 1.08%) for Al-Soofi and Dwaikhla Kaolin, respectively. It was found that the brightness of these clays was improved by the biobleaching process from 47.61 to about 59.59; for Al-Soofi Kaolin and from 63.23 to about 69.08, for Dwaikhla Kaolin. It is also worth to mention that wet sieving of Dwaikhla Kaolin on 45 μ sieve improved the brightness up to 70.98.

قصر لون أطيان الكاؤولين من موقعي وادي الصوفي ودويخلة في الصحراء الغربية العراقية
بالطرق البايولوجية لاستخدامها في الصناعات الورقية

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المستخلص

تم في هذه الدراسة قصر لون أطيان الكاؤولين لموقعي دويخلة ووادي الصوفي في الصحراء الغربية العراقية بايولوجياً باستخدام الفطر *Aspergillus niger*، حيث أمكن تقليل نسبة أكاسيد الحديد والتيتانيوم المسببة لتلون هذه الأطيان، إذ تم خفض محتوى الـ Fe_2O_3 في كاؤولين وادي الصوفي بنسبة 28.31% (من 2.19% إلى 1.57%)، بينما خفض هذا المحتوى في كاؤولين دويخلة من 1.61% إلى 1.10% ونسبة تقدر 31.25%، من جهة أخرى تم خفض محتوى الـ TiO_2 في هذه الأطيان بنسبة 33.6% و 27.0% (من 1.16% و 1.48% إلى 0.77% و 1.08%) لكاؤولين وادي الصوفي ودويخلة على التوالي. إن عملية القصر الحيوي أدت إلى تحسين درجة السطوع لهذه الأطيان من 47.61 إلى 59.59 لكاؤولين وادي الصوفي ومن 63.23 إلى 69.08 لكاؤولين دويخلة. ومن الجدير بالذكر أن الغربلة الرطبة لكاؤولين دويخلة على منخل 45 μ قد أدت إلى تحسين درجة السطوع لتبلغ 70.98.

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INTRODUCTION

Dwaikhla and Al-Soofi Kaolins are among the major kaolin deposits in Iraq. These deposits have brightness value of about 47.61 and 63.23, respectively. The low brightness prevents these clays from utilization by paper industries. Paper industry requires a range of brightness of (79 – 83.3) and (83.5 – 85.5) for filler and coating materials, respectively (Luciana *et al.*, 1996). "The kaolin is comprised of kaolinite mineral, which is hydrated aluminum silicate ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$). The major discoloring of the clays is due to the presence of iron oxides and hydroxides (usually Fe^{+3} forms) that commonly are precipitated or adsorbed on the clay surfaces, or admixed as a separate phases, during the arising of these clays from the stones resulted from mechanical and chemical factors depending on the climatic and geological conditions" (Murad, 1987 and Stucki *et al.*, 1988).

Kaolin clays are essential source for many industries; such as ceramic, paper, paint and fillers (Asmatulu *et al.*, 2002). One of the most important factors that affect the utilization of Kaolin clay in paper industries is its brightness, which is mostly affected by contaminants, such as iron oxides that is deposited on the kaolin particles during its formation, which make much of the kaolin unsuitable for the industries mentioned above (Hosseini *et al.*, 2007). According to the aforementioned reason, the quality of kaolin is measured in terms of iron content (Lee *et al.*, 2002). Therefore, for most modern industrial applications, kaolin must be extensively refined and processed to enhance commercial characteristics. In paper industry, which is the main consumer, the most important specification of kaolin is the brightness. Generally, a brightness of 79 – 83.5 is required for filler and 83.5 – 85.5 for coating. However, the process employed for improving the brightness of mineral product can be classified as physical and chemical ones, or both (Luciana *et al.*, 1996).

It has been recently recognized that a large population of microorganisms inhabits diverse subsurface environments including sub-sea floor sediments and basement rocks, continental sedimentary rocks, ancient salt deposits, aquifers in igneous terrestrial rocks, and caves (Krumholz, 2000). Claystone formations represent a unique subsurface microbial habitat. Due to their very fine particle size, claystone when are not exposed (in subsurface) have low hydraulic conductivity and subsequently the rate of microbial metabolism is extremely slow. Microorganisms are generally larger than clay mineral particles and it has been suggested that microbial populations present in clays were trapped there during deposition of the clay layers. If this is the case, the micro organisms may have survived from the time of original clay sedimentation tens of millions years ago (Lawrence *et al.*, 2000). The Georgia Kaolins of the United States were for long thought to have been deposited as unusual white nearly mono-mineralic clays, but recent evidence indicates that they were deposited as typical dark organic muds in a deltaic sequence of interlayered sand and clay swamp and marsh flood plain clastics. It was suggested that, what is now nearly pure kaolin clay, has been greatly whitened by weathering during subsurface burial, and that bacterial action has played a critical role in removal of iron and organic matter (Evgenya *et al.*, 2005). The key criteria for the industrial use of Georgia's commercial white kaolin deposits are the low contents of discoloring iron oxide, titanium oxide and iron sulfide impurities, and small dark organic carbon contents. The iron is present in kaolin as cream to tan-tinted kaolin in the form of Fe^{+3} oxides and hydroxides (Hematite and Goethite) and as a structural occupation. In gray kaolins, which have not yet been oxidized, iron is present mainly as pyrite. Gray kaolins have a relatively large dark organic carbon content, which causes their distinct gray or dark brown appearance (Evgenya *et al.*, 2005).

The aim of this work is the removal of undesirable impurities such as Fe_2O_3 and TiO_2 from Dwaikhla and Al-Soofi Kaolins using biological treatment. Thus, improves the brightness of the kaolin to match the specifications of filler materials for paper industry.

▪ Previous Works

Different physical, chemical and microbiological techniques were developed for the removal of ferric or hydrated iron oxides present in clays. Traditional techniques, such as magnetic separation, froth flotation, selective flocculation, size separation (by hydro cyclones), and leaching were used for removal of iron impurities from clays (Štyriaková and Štyriak, 2000). However, the removal efficiency of iron was low; because the iron in kaolin may be tightly adsorbed or it may be in complex form (Lee *et al.*, 2002), also it may occupy the vacant spaces between the tetrahedrons and octahedrons of the kaolinite (Jassim *et al.*, 2009) that is not easily separated by the traditional methods.

The chemical methods are divided into two routs: The first rout includes the treatment of kaolin clays with mineral acid, while the second rout is represented by treatment with reducer materials. Poorly crystalline or more readily soluble iron oxide phases can be removed using various methods; such as ammonium oxalate or dilute HCl extraction (Stucki *et al.*, 1988). Complete separation or removal of iron oxide phases by chemical means without in some way altering the remaining phase is unlikely (Manceau *et al.*, 2000). Furthermore, the clay structure collapses during reduction in response to the reduction of structural iron (Kostka *et al.*, 1999). Most of the industries have employed potent chemical reductants such as dithionate or hydrazine to remove iron impurities, but these chemicals are not the cause of iron reduction in nature (Kostka *et al.*, 1999). Chemical methods are usually suitable for achieving higher extent of iron removal, but at the same time they are more expensive, the operating conditions are very complicated, and the process are environmentally dangerous (Luciana *et al.*, 1996 and Hosseini, 2007).

Heterotrophic microbial (bacteria and fungi) leaching has been studied for many industrial minerals, and demonstrated that iron could be removed from quartz sand by using microbial leaching to remove ferruginous compounds (Groudev *et al.*, 1985 and Groudev, 1987). Microbial removal of iron from clays and improvement of kaolin by the action of metabolic products has also been demonstrated (Groudev *et al.*, 1989). The attack on minerals is due to the effect of produced organic acids, amino acids and other metabolites that are excreted into the culture medium, heavy metals are dissolved by direct displacement of metal ions from the ore matrix by hydrogen ions and by the formation of soluble metal complexes and chelates. These microorganisms require an organic carbon source for growth and energy supply, not taking any advantage from mineral degradation. The heterotrophic microorganisms' ability to dissolve metals from solid materials could in some cases be successfully used for improvement of quality of mineral raw materials, such as kaolin clays and sands. Because microbial leaching is rather simple to perform, low in capital and energy costs and free of environmental pollution, this technique is considered to be of great importance for future material treatment, principally applied to non-sulfide ores and minerals (Cameselle *et al.*, 2003).

MATERIALS AND METHODS

▪ The Clay Samples

Raw kaolin clay samples of 20 Kg (each) brought from Dwaikhla and Al-Soofi locations were subjected to crushing and screening to pass 8 mm sieve opening. Then quartered and divided to obtain identical samples of 1 Kg, each. Representative Samples of Dwaikhla and Al-Soofi Kaolins were then drawn for chemical analysis. Brightness measurements were executed using Hunter lab colorimeter (color flax). The results of the chemical analysis and brightness are shown in Table (1).

Table 1: Chemical analysis of Dwaikhla and Al-Soofi raw Kaolins

Sample	Chemical Composition (%)					Brightness
	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	TiO ₂	L.O.I	
Al-Soofi Kaolin	53.48	2.19	29.05	1.16	11.42	47.61
Dwaikhla Kaolin	49.39	1.61	33.49	1.48	11.84	63.23

▪ Biological Media and Chemicals

- Yeast extract agar: for semi-solid media cultivation of *Aspergillus niger*
- Sugars: glucose, sucrose and date extract; used as carbon source for mold growth
- Nitrogen source: ammonium nitrate, ammonium chloride and urea
- Hydrochloric acid: for pH-adjustment of culture solution
- Antiseptics: ethanol and detol for sterilization
- Nutrition complement: used with minor doses like potassium chloride, magnesium sulfate, and dihydrogen potassium phosphate

▪ Physical Pretreatments

Two physical techniques were used for preliminary upgrading the kaolin clays of Dwaikhla and Al-Soofi valley. The first technique includes; sieving of 20% solid slurries of these clays on 45 μ sieve opening; to remove + 45 μ portion. This process allows obtaining low quartz content clay, which will improve the physical as well as chemical properties of kaolin clay, especially for paper industry. The other technique includes; grinding of the clay to pass through 0.5 mm sieve opening, then the clay was subjected to dry high intensity magnetic separation using (8,16.5 and 18) kilogauss to remove the magnetic fraction, which is rich with Fe₂O₃.

▪ Biological Treatments

These treatments included two stages:

—**First Stage:** This stage is related with isolation, activation, inoculation and incubation of the mold *Aspergillus niger*. Parameters that affect on the final pH; such as fermentation (incubation) temperature and initial pH were fixed at 30°C and pH 7.0, respectively according to the optimum value obtained from a previous study (Mustafa, 2008).

—**Second Stage:** This stage concerns with the biobleaching process, where clays are mixed with culture solutions. In this stage, many parameters were studied.

The parameters that were studied for both stages of the biological treatment are as follow:

Three concentrations of Glucose (50, 100 and 50) gm/l were used to identify the best concentration needed for optimum mold growth; while two temperatures were used (25 and 100)° C to achieve the optimum temperature for bioleaching

Also the experiments were performed using three solid percentages (1, 2 and 5%) of – 45 μ friction kaolin bioleaching experiments were conducted with three pH values (0.5, 7.0 and 12), where the pH was adjusted by concentrated HCl. Whereas another experiment was done with HCl concentration of 5% .

Furthermore, several experiments were achieved to found the best nitrogen sources for the optimum growth of the mold, which in turn means optimum secretion of organic acids that are responsible for the bioleaching; for this aim two types of nitrogen source were used (ammonium nitrate and urea).

RESULTS AND DISCUSSION

▪ Physical Pre-treatment

– **Wet Sieving:** This process was used to reduce the free quartz grain content in the kaolin clay, as well as to improve its brightness. Table (2) shows the chemical analysis of the – 45 μ clay portion.

Table 2: Chemical composition of the – 45 μ portion of kaolin clay

Kaolin clay sample	Chemical Composition (%)					Recovery (%)	Brightness
	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	TiO ₂	L.O.I		
Al-Soofi Kaolin	49.29	2.16	28.96	1.17	11.46	94.6	51.98
Dwaikhla Kaolin	48.49	1.59	33.77	1.48	12.11	95.6	70.98

From the results shown in Table (2), it is obvious that the brightness of Dwaikhla Kaolin is largely improved after wet screening on – 45 μ sieve. Nevertheless, a little decrease in Fe₂O₃ content was obtained, only by 1.24%, while TiO₂ content remained constant. The same results were obtained in Al-Soofi Kaolin, while brightness increased by about 4 digits. However, the brightness of the two clays is still far from that required for fillers used in paper industry.

– **Magnetic Separation:** Many experiments have been conducted using magnetic separation; by subjecting the – 0.5 mm portion of Al-Soofi Kaolin to high intensity magnetic separation to lower the Fe₂O₃ contents, and to improve its brightness. The results are shown in Table (3).

Table 3: Chemical composition of non-magnetic portion of Al-Soofi Kaolin at different intensities

Magnetic Intensity	Chemical Composition (%)					Brightness
	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	TiO ₂	L.O.I	
8 Kilogauss	54.8	2.15	29.42	0.99	11.24	52.18
16.5 Kilogauss	55.0	2.13	28.96	0.96	11.36	52.98
18 Kilogauss	55.02	2.10	28.85	0.92	11.53	54.12

It can be seen from the results illustrated in Table (3), that magnetic separation had lowered the Fe₂O₃ by 13.5% and TiO₂ by 20%. This was reflected by brightness increasing by 13.5%, to gain more improvement in the brightness; more Fe₂O₃ and TiO₂ removal must be achieved and thus another approach have to be applied.

▪ Biological Treatments (Bio-bleaching)

Several experiments have been conducted using culture solutions obtained from fermentation (incubation) of *Aspergillus niger* in the growth media. These media have several components that have vital role in the growth of this mold. Some of these components are organic and others are inorganic. The most important organic component is sugar, which is the carbon source, as well as precursor for the organic acid (mainly citric and oxalic acids) that are essential for the leaching of iron, beside its capability to chelates Fe⁺³ ions preventing them from re-adsorption on the clay particles once again.

–**Effect of Carbon Source:** Three sugars were tested to evaluate their effect on the removal of discoloring minerals; such as Fe_2O_3 and TiO_2 . These sugars include Glucose, Sucrose and Date Extract. The results are shown in Table (4).

Table 4: Effect of carbon source on the biobleaching of kaolin

Clay Sample	Carbon Source	Chemical Composition (%)					Brightness
		SiO_2	Fe_2O_3	Al_2O_3	TiO_2	L.O.I	
Al-Soofi Kaolin	Glucose	49.56	1.93	27.82	0.88	15.44	53.29
	Sucrose	51.44	2.03	27.75	1.0	15.04	49.84
	Date Extract	52.77	2.03	30.13	0.93	12.50	50.41
Dwaikhla Kaolin	Glucose	46.66	1.10	29.14	1.19	18.97	69.08
	Sucrose	50.68	1.19	31.93	1.19	12.46	56.19
	Date Extract	51.38	1.18	31.34	1.08	11.84	60.32

From the results tabulated in Table (4), it can be easily recognized that the biobleaching of Dwaikhla Kaolin had lowered the brightness of the product, as compared with that of the feed (– 45 μ portion). Although, Fe_2O_3 and TiO_2 were highly reduced via biobleaching, brightness was decreased. This may be attributed to the opaque color developed during fermentation process, especially when sucrose or date extract was used as carbon source. On the other hand, the brightness of Al-Soofi Kaolin was slightly increased after biobleaching by 11.9%; from 47.61 to 53.29 using glucose as a source of carbon. Also iron and titanium oxides were decreased to (11.8 and 19.8) %, respectively. According to the results, biobleaching of Dwaikhla Kaolin seems to be not useful for brightness improvement. While the brightness of Al-Soofi Kaolin responded positively to the biobleaching process, particularly when Glucose was used as a source of carbon.

–**Effect of Temperature of Biobleaching:** To investigate the possibility of Al-Soofi Kaolin biobleaching at a lower temperature, an experiment was carried out at room temperature (25° C). The results in Table (5) pointed out that the efficiency of biobleaching was lowered by high extent; when it was carried out at room temperature (25° C). This is resulted from removal of small amounts of Fe_2O_3 and TiO_2 , as compared with the achieved results at boiling temperature (100° C).

Table 5: Effect of temperature on the biobleaching of Al-Soofi Kaolin

Temperature	Chemical Composition (%)					Brightness
	SiO_2	Fe_2O_3	Al_2O_3	TiO_2	L.O.I	
100° C	49.56	1.93	27.82	0.88	15.44	53.29
25° C	50.20	2.10	28.77	1.10	13.76	49.19

–**Effect of Solid Percentage:** It is worth to mention that most of the carried out researches concerning biobleaching of kaolin; are in a range of solid percentage of (2 – 6) % (Cameselle *et al.*, 2003; Hosseini *et al.*, 2007). In this study, solid percentage of 5% was used, because it gives sufficient amounts of product for chemical analysis plus brightness.

–**Effect of pH on Biobleaching:** To understand the role of pH on the biobleaching efficiency, pH of biobleaching was adjusted to certain values of 0.5, 7.0 and 12 that resemble the acidic, neutral and alkali conditions. Another experiment was carried out without pH adjustment but with the addition of 15 ml of 5% HCl to the medium before biobleaching.

The best results in (Table 6) were achieved under highly acidic condition. The lower the pH the more removal of Fe_2O_3 and TiO_2 was achieved. This is attributed to the leaching effect of available H^+ ions in the acidic condition, where citric and oxalic acids acted as a reducer and chelates for Fe^{+3} and Ti^{+4} .

Table 6: Effect of biobleaching modification

Type of Modification	Chemical Composition (%)					Brightness
	SiO_2	Fe_2O_3	Al_2O_3	TiO_2	L.O.I	
pH-Adjustment (0.5)	50.96	1.67	29.69	0.41	12.77	58.70
pH-Adjustment (7.0)	51.06	2.10	28.29	0.60	13.19	54.16
pH-Adjustment (12)	53.56	2.10	29.06	0.60	10.95	53.50
5% HCl Addition	50.98	1.71	29.07	0.58	14.06	56.28

–**Effect of the Nitrogen Source:** Ten experiments have been conducted to select the best nitrogen source for biobleaching. These experiments were carried out at different times, (2, 3 and 4) hours were used to optimize the biobleaching efficiency. The results are presented in form of brightness versus biobleaching time and are shown in Fig. (3).

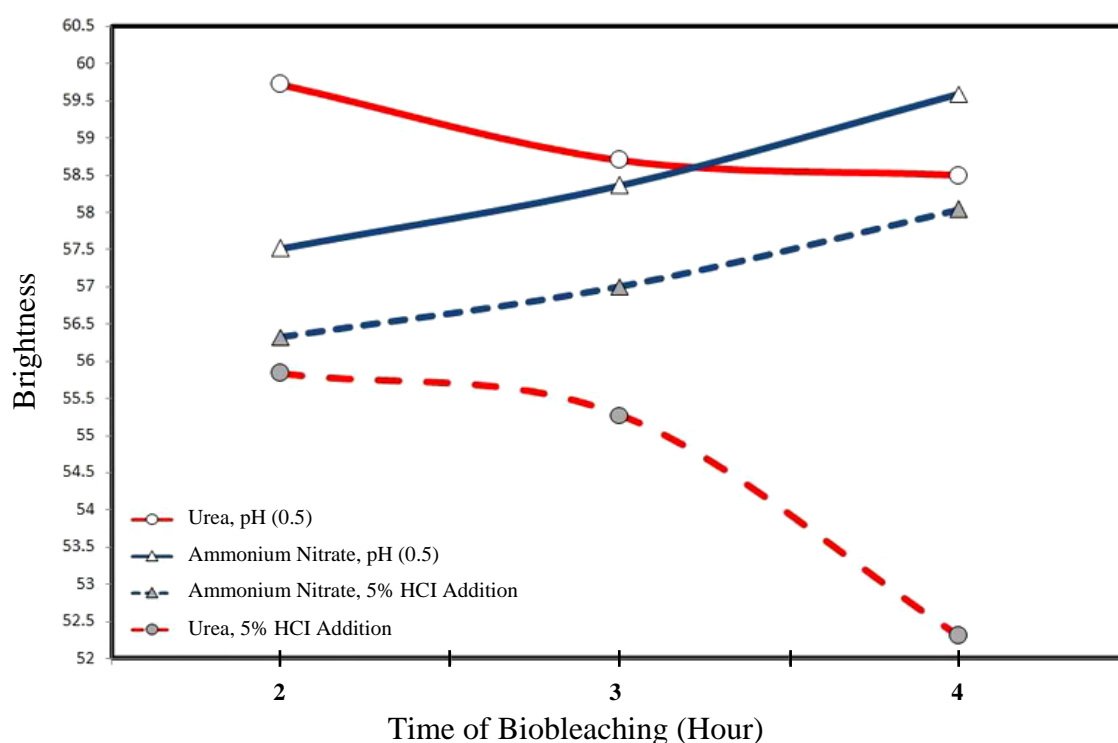


Fig.3: Effect of nitrogen source and time on the biobleaching process

According to the results shown in Fig. (3), it can be observed that each nitrogen source has a unique behavior during biobleaching. Accordingly, when ammonium nitrite is used as a source of nitrogen and pH-adjusted to 0.5, the brightness was increased with the increasing in time from 2 to 3 and up to 4 hours. Similar behavior was observed, with 5% HCl addition instead of pH-adjustment, the only difference that recognized was the lower brightness of the product.

Furthermore, when urea was used as a nitrogen source, a considerably reverse relationship between brightness and biobleaching time occurred. As time was increased from 2, 3 and finally 4 hours, brightness was decreased and also in this case, a pH-adjustment to 0.5 gave a product with higher brightness than the 5% HCl addition. This behavior may be caused due to the degradation of urea by HCl with time, and assisted by higher temperature of biobleaching, because most organic compounds degrade by higher temperatures or mineral acid; or by both of them. However, brightness of Al-Soofi Kaolin showed a considerable improvement; up to 59.59, but it is still far below that required for filler clay in paper industry, which is in the range of 79 – 83.

The results of chemical analyses for selective products with physical and biological treatments of Al-Soofi and Dwaikhla Kaolin, which have achieved the highest brightness in this work are shown in Table (7).

Table 7: Chemical composition for selective biobleaching products

Kaolin Clay Sample	Chemical Composition (%)					Brightness
	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	TiO ₂	L.O.I.	
Al-Soofi (– 45μ)	49.29	2.16	32.20	1.17	11.46	51.98
Dwaikhla (– 45μ)	48.49	1.59	33.77	1.48	12.11	70.98
Al-Soofi (Biobleached)	57.78	1.32	27.82	0.77	10.95	59.59
Dwaikhla (Biobleached)	46.66	1.10	29.14	1.19	18.97	69.08

CONCLUSIONS

According to the biobleaching test results for Dwaikhla and Al-Soofi Kaolins, the following conclusions can be stated:

- The brightness of Dwaikhla Kaolin enhanced by 12.2%; when slurry of kaolin passes through 45μ sieve opening. The brightness obtained was about 70.98, comparing with 63.23 of the raw clay.
- Although iron and titanium oxides are removed by 31.25% and 27%, respectively via biobleaching, the brightness was decreased. Therefore, this process cannot be recommended for brightness enhancement for Dwaikhla Kaolin.
- Although appreciable amounts of Fe₂O₃ and TiO₂ can be removed through biobleaching from Al-Soofi Kaolin, and accordingly, brightness can be improved up to 25.1%, the achieved brightness was still far from that required for filler clay in paper industry.
- Biobleaching was not useful method for increasing Dwaikhla Kaolin brightness. This, may be attributed the adsorption of the opaque color enveloped by the fermentation of sugar in the culture medium.

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