International Journal of Advanced Chemistry Research 2025; 7(10): 27-29

International Journal of Advanced Chemistry Research

ISSN Print: 2664-6781
ISSN Online: 2664-679X
NAAS Rating (2025): 4.77
IJACR 2025; 7(10): 27-29
www.chemistryjournals.net
Received: 17-07-2025
Accepted: 21-08-2025

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Influence of calcination temperature and acid concentration on the properties of gold pearlescent pigment using biotite

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DOI: https://www.doi.org/10.33545/26646781.2025.v7.i10a.320

Abstract
The paper proposes a novel method of preparing gold pearlescent pigments using biotite instead of muscovite. This method has a lower calcination temperature of 100 °C and no phase transformation process of titanium dioxide is required. As a result, it is possible to produce high quality pigments with energy saving and cost reduction.

Keywords: Gold pearlescent pigment, biotite, ferric chloride, energy saving

Introduction
The structure of biotite is K2(Fe, Mg)SiAl2O2(OH, F) and K2(Fe, Mg)AlSiAlO2(OH, F). In particular, biotite containing a large amount of Fe is called ferro mica and exhibits a black color.

Gold pearlescent pigments are obtained by nanocoating of iron oxide on mica titanium and its starting materials are muscovite [1-3]. The quality of mica titanium requires good quality muscovite. In general, muscovite is regionally heterogeneous, especially in the case of talc content in muscovite. There are not many kinds of white mica that can be used for the preparation of mica titanium. However, biotite does not differ significantly from reservoir to reservoir. Examples of gold pearlescent pigments prepared by direct calcination of phlogopite have been reported [4, 5].

In the case of preparing pearlescent pigments of different colors, including gold pearlescent pigments, using muscovite as starting material, mica titanium should be prepared first [6-8]. The purity of titanyl sulfate as a deposition material is important for the preparation of mica titanium, and the high temperature required to convert the deposited titanium dioxide into rutile forms increases energy consumption and consequently the cost of the pigment.

Therefore, we have made a research to produce high-quality gold pearlescent pigment at low calcination temperature with biotite and ferric chloride as starting materials and established the production process.

Materials and Method
Raw materials
The raw materials used are biotite (raw ore), sodium hydroxide (industrial grade), and ferric chloride (industrial grade).

Method
The biotite was ground to a size of 3~5 mm, calcined in a kiln for a certain time and then cooled naturally. After grinding in a vibrating mill for 4h, the flotation and classification were carried out by a fine-scale classifier.

Mica crushes in the range of 30~60 µm was acid washed and dried. The dried mica powder was suspended in a solid-liquid ratio of 1:12 and coated with iron oxide for 1.5h with dropping ferric chloride. The pH was maintained at 4.2 with 0.1mol/L NaOH solution during the dropping of ferric chloride. After aging for 30min, filtering, washing, drying, and coating with nanosilica to complete the preparation.

Results and Discussion

Chromatic changes of biotite with calcination temperature

The biotite used as a raw material is a black mica with a high iron content.

When calcined, it was changed in different colors with temperature. The calcination time was 3h. The color changes of biotite with calcination temperature is as follows:

Table 1: Chromatic changes of biotite with calcination temperature

Temperature, °C	Chromatic changes
400	Black
500	Grey
600	Grey
700	Light yellow
800	Light yellow
900	Light yellow

The color does not change from 700 °C because the iron content in biotite is oxidized enough to convert to oxide. Considering the color change with time at 700 °C, the calcination temperature of biotite was 700 °C, and the color was no longer changed at 3h. The milled biotite was calcined at 700 °C for 3h, cooled naturally and then ground for 4h in a vibrating mill. The ground mica suspension was floated using triethanolamine as a flotation reagent.

The classification was done using a hydro-cyclone precision classifier with a branch tube. The graded mica shall be washed, dried and sent to acid treatment process.

Effect of acid concentration on the gloss of gold pearlescent pigment

The activation treatment of graded mica (30-60 um) has a great influence on the final product properties. Without activation, the gloss decreases and the nanometal oxide coating become difficult. Hence, the effect of acid and alkali treatment on the gloss of gold pearlescent pigment on calcined and graded biotite was investigated.

The alkali treatments on biotite showed no significant effect. Thus, different concentrations of acid treatment were carried out on the calcined biotite. The following table shows the change in the gloss of gold pearlescent pigment with the hydrochloric acid concentration. The acid treatment temperature was 80 °C and the time was 1.5h. After the acid treatment, the final product is obtained by washing, filtering, coating with iron oxide with the addition of ferric chloride and coating with nanosilica. The iron oxide coating amount was 15%.

Table 2: Effect of acid concentration on the properties of gold pearlescent pigment

HCl concentration, %	Gloss, %
5	50
10	56
15	82
20	85
25	85.8
30	85.9

No significant change in color was observed from the HCl concentration above 20%. It is expected that the acid-soluble materials were fully released from the calcined biotite surface.

Comparison of energy consumption

The energy consumption of the process of producing gold pearlescent pigment with muscovite and biotite as starting materials, respectively, was compared. Comparing the firing temperature of the raw material, the case of biotite is 100°C lower than that of muscovite.

In the case of muscovite as the starting material, a high temperature of 900 °C is required to transfer titanium dioxide to the rutile form. But, in the case of biotite, there is no the calcination process. The results are given in Table 3 below.

Table 3: Comparison of energy consumption

Process	Calcination temperature, °C	
	Muscovite	Biotite
Raw material calcination temperature, °C	800	700
Titanium dioxide phase transition temperature, °C	900	-

Conclusion

Gold pearlescent pigment was prepared by calcining biotite at 700 °C for 3h and coating iron oxide. There are no significant differences compared to gold pearlescent pigments prepared from mica titanium as the starting material.

We established a method of producing gold pearlescent pigment with energy saving and low cost and built a process. It is expected that the effect of gold pearlescent pigments will be better if the surface of the calcined biotite is first coated with titanium dioxide and then coated with other metal oxides such as iron dioxide.

Further studies are needed to prepare pearlescent pigments of different colors by first coating the calcined biotite surface with titanium dioxide and coating the multilayer nanometal oxides on it.

Acknowledgements

The authors would like to express their gratitude to anonymous reviewers for their constructive comments.

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