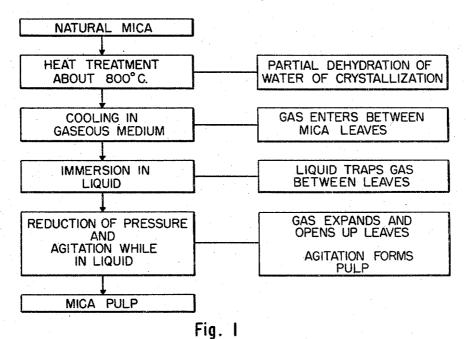
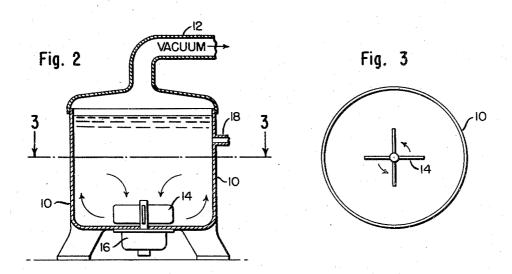
PROCESS FOR PRODUCING MICA PULP

Filed June 12, 1951





INVENTOR.

Autorius Tran Grovges Bovelest

BY

Kenway, Jenney, Us His

and Hildreth, atlys.

1

## 2,709,158

## PROCESS FOR PRODUCING MICA PULP

Antoine Jean Georges Bouchet, Paris, France, assignor to Samica Corporation, Dover, Del., a corporation of

> Application June 12, 1951, Serial No. 231,271 4 Claims. (Cl. 252-378)

This invention relates to the production of mica pulp 15 and more particularly to an improved process for producing a mica pulp capable of forming mica paper and molded articles wherein mica flakes cohere without necessity for bonding agents. The mica pulp produced by the process of this invention is elsewhere described 20 in Letters Patent No. 2,549,880 to Jacques Jules Bardet.

The process described in the above-mentioned Bardet Patent No. 2,549,880 consists essentially in first heating a batch of blocks and pieces of natural mica to a temperature of about 800° C. for sufficient time to eliminate 25 therefrom a portion of the hydroxyl ions in the mole-This heat treatment had the effect of slightly changing the visual appearance of the mica and also of expanding the pieces of mica. While the mica was still hot, it was quenched in a concentrated soda solution, 30 following which the soda was drained off and the mica then immersed in a solution of sulphuric acid. This sequence of steps brought about a partial softening of the mica and a cleavage of the leaves or scales of the mica pieces. The soda entered the interstices between 35 the mica leaves during quenching, and thereafter the sulphuric acid reacted with the soda to produce gas between the said leaves and gradually to force them apart. After the acid bath, the mica was in the form of substantially finely divided, thin flakes with a few blocks 40 remaining in an expanded and exfoliated condition. The next steps in the original process included washing out the salts formed by the acid soda reaction, and thereafter introducing the mica into an agitator which completed the final disintegration of remaining pieces. After 45 agitation, the mica was in an extremely finely divided state of suspension resembling a pulp and could either be dried into cake form for shipment or else employed immediately in a paper-making or molding process.

tain disadvantages inherent in its practice. In the first place the use of sulphuric acid was somewhat objectionable as requiring special acid-resisting equipment. Secondly, the process resulted in the formation of large and consequently tended to pollute the adjoining rivers into which the effluent was discharged. Thirdly, and perhaps the most significant, disadvantage in the original process was the fact that it required numerous steps including repetitive removal of batches of mica from baths, 60 transportation of the same, changes of liquid media, etc. This excessive handling of the mica was regarded as undesirable because of certain minor losses of mica from step to step, and also because of the added labor costs incident to each step of the process.

In searching for a more efficient and simpler process, the previously known methods for treating mica were of little avail. It must be borne in mind that after heat treatment to about 800° C. and partial dehydration, mica becomes noticeably limp, and for that reason the conventional processes of grinding, rubbing, pounding, or other violent treatments of mica for separation thereof

which converted the mica into a dust could not be employed to produce the pulp described in the Bardet Patent No. 2,549,880. Such violent working of the mica after the said heat treatment did not provide a product fit for formation into commercial sheets or articles.

Therefore it is an object of this invention to provide a process for producing mica pulp, which may be carried out with a minimum number of steps and without constant shifting of batches of mica from one liquid 10 medium to another. It is a further object of my invention to provide a process for producing mica pulp, which is more suited for continuous operation and in which efficiency may be improved by permitting recovery of heat by the elimination of quenching steps. An additional object of my invention is to provide a process which accomplishes the foregoing objects without subjecting the mica to violent or destructive action and thereby permits the production of a good quality of mica pulp.

In the accomplishment of these and other objects of my invention, in a preferred embodiment thereof, I commence by heat treating natural mica to a temperature of about 800° C. and for sufficient time to eliminate approximately one-half of the hydroxylions therefrom and to expand the individual blocks of mica. Following this heat treatment I permit the mica to cool, at which time the retained heat in the mica may be partly recovered by conventional heat exchange mechanism. Following cooling, I introduce the heat treated mica into a liquid medium which may be the same liquid medium employed in the final paper-making or molding stages for the pulp. The said medium is preferably water, but may be other liquids or emulsions provided they do not inhibit agglomeration of the mica flakes during the formation of sheets or other articles. While the mica is in this medium I reduce the pressure over the surface of the said medium to expand the gas retained between the leaves of the mica and thereby promote exfoliation of the same. Simultaneously I also mildly agitate the mica in the same liquid medium thereby bringing about a mechanical exfoliation and disintegration of agglomerates along with the exfoliation which is caused by the expansion of the gas between the mica leaves.

To assist in an understanding of my invention, I have provided the accompanying drawings in which:

Fig. 1 is a flow diagram of the process of my inven-

Fig. 2 is a view in side elevation of the vat adapted While this process was satisfactory, there were cer- 50 for low pressure and agitation of mica in the liquid medium; and

Fig. 3 is a sectional view along the lines 3-3 of

It will be understood that permitting the heat treated quantities of salts which had to be totally eliminated 55 mica to cool in the air or other gas for a substantial time permits air to penetrate between the mica leaves which have been partly opened up by the heat treatment. Thereafter when the mica is immersed in the liquid medium, a large part of the air between the leaves remains there and its expansion is accomplished by reducing the pressure of the said medium to a value substantially below the pressure of the air between the leaves. This may be done either by subjecting the mica to high pressure in a gaseous medium prior to immersing the mica in the liquid, and thereafter releasing the pressure; or else it may be done by maintaining the mica under atmospheric pressure after cooling and by then drawing a vacuum over the liquid medium after the mica has been immersed therein.

A word of caution is deemed necessary with respect to the temperature to which the mica is heated in order to obtain partial dehydration. Although it may be that

different types of mica require slightly different temperatures, the figure of 800° C. is regarded as the best approximation. However, the volume of the particular batch of mica undergoing treatment will to some extent determine the amount of heat supplied to it from 5 the exterior. It must be remembered that mica is an excellent thermal insulator; consequently it will require a longer time for heat to penetrate the interior of the mass of the batch, the time increasing with the size of the mass. At the same time if excessive heat is applied 10 to the interior of the mass there is danger of complete dehydration of the surface portions thereof which results in complete pulverization of the said surface portions and consequently a poor pulp. With the figure of 800° C. as the average optimum, those skilled in the 15 art will readily recognize that the desired dehydration may be obtained either by excessive heating for a short period of time, the time being insufficient completely to dehydrate a significant portion of the mica, or the heat treatment may be carried out at a lower temperature 20 for a longer period of time to obtain thorough penetration of the heat into the interior of the mica without completely dehydrating any of it. This is not to say that the desired dehydration could be obtained by heating the mica for an indefinite period at, say, 400° C. because 25 it has definitely been established that a temperature in the general neighborhood of 800° C. is required if the desired result is to be obtained. However, heating for several hours at 750° C. will often prove the practical equivalent of heating for a few minutes at 850° C. For the 30 sake of convenience we have chosen the expression "approximately 800° C." to describe the heating step within the general limits of time and temperature as discussed herein.

Water is definitely the preferred liquid medium for 35 carrying out my invention. However, I have found that other aqueous solutions and emulsions of, for example, melamine formaldehyde, have been satisfactory. foliation may be accomplished in still other liquid mediums such as gasoline, benzene, anhydrous alcohols, 40 and the like, where the end use of the pulp may be for purposes other than making paper. However, if such latter-mentioned mediums are employed, and thereafter it is desired to prepare the pulp for making mica paper, the said medium need only be washed out and replaced 45

by an aqueous medium.

The mechanical agitation of the mica pulp is carried out in a large circular vat 10 adapted for the previously described pressure differential as indicated at 12 where a vacuum is drawn and provided with a propeller 14 driven by a motor 16. The propeller 14 circulates the mica from a point near the bottom and center of the vat The fully exfoliated mica enters into suspension and becomes distributed throughout the water in the nearer the propeller at the bottom. The pulp in suspension is drawn off from the top of the vat at 18. In this manner the lighter and more fragile mica flakes do not receive destructive agitation treatment.

From the foregoing description it will be seen that 60 the process of my invention may be carried out with a minimum of steps. Agitation and exfoliation are accomplished simultaneously without excessive treatment, and the constant changing from one medium to another

has been eliminated because only one liquid medium is necessary.

Various minor variations of the process of my invention will now be apparent to those skilled in the art and therefore it is not intended to confine the invention to the precise form of the preferred embodiment herein shown but rather to limit it in terms of the appended claims.

Having thus disclosed and described an illustrative process of my invention, what I claim as new and desire to secure by Letters Patent is:

- 1. A process for producing mica pulp comprising the steps of heating mica to approximately 800° C. for sufficient time partially to open up the leaves thereof, introducing a substantially cooler gas into the interstices between said leaves formed by said heating step, confining said gas between said leaves by immersing the same in a liquid, and thereafter further exfoliating said mica in said liquid by agitating the same and by expanding said confined gas by lowering the pressure on the surface of said liquid to a value substantially lower than the pressure of said gas between said leaves after said immersion.
- 2. A process for producing mica pulp comprising the steps of heating mica to approximately 800° C. for sufficient time partially to open up the leaves thereof, introducing air at substantially room temperature into the interstices between said leaves formed by said heating step, confining said gas between said leaves by immersing the same in a liquid, and thereafter further exfoliating said mica in said liquid by agitating the same and by expanding said confined air by lowering the pressure on the surface of said liquid to a value substantially lower than the pressure of said air between said leaves after said immersion.
- 3. A process for producing mica pulp comprising the steps of heating mica to approximately  $800^{\circ}$  C. for sufficient time partially to dehydrate the same and to open up the leaves thereof, introducing air at substantially room temperature into the interstices between said leaves formed by said heating step, confining said gas between said leaves by immersing the same in a liquid, and thereafter further exfoliating siad mica in said liquid by agitating the same and by expanding said confined air by lowering the pressure on the surface of said liquid to a value substantially lower than the pressure of said air between said leaves after said immersion.
- 4. In a process for producing mica pulp, the combination of steps comprising, opening up and partially dehydrating mica by heating it to approximately 800° C., thereafter introducing gas between the leaves of said mica at a temperature and pressure suitable for subsequent expansion of said gas while said mica is immersed in a liquid, and then further exfoliating said mica by simulvat, while the still partially agglomerated cakes remain 55 taneously agitating the same in a liquid and expanding said gas.

## References Cited in the file of this patent UNITED STATES PATENTS

686,929	Heard et al.	Nov.	19,	1901
2,549,880	Bardet	_ Apr.	. 24,	1951