[1,2] A. S. Kucuker,
[2,3] A. Kara,
[1] K. Kayacı
[4] S. Gerl

[1] Termal Seramik San. ve Tic. A.S., Bilecik, TURKEY
[2] Anadolu University, Dept. of Material Sci. and Eng., Eskisehir, TURKEY
[3] Ceramic Research Center A. S., Eskisehir, TURKEY
[4] Maschinenfabrik Gustav Eirich GmbH & Co. KG., Hardheim, GERMANY

Cost Effective Slurry Preparation in Porcelain Tile Production

Abstract

During ceramic tile production (wall, floor and porcelain stoneware), wet milling process for the preparation of ceramic slurries is widely used. The currently preferred wet grinding machines are discontinuous and/or continuous ball mills, where grinding occurs when a particle is crushed between two pieces of grinding media. Ball milling is, however, not a cost effective method, especially in preparation of porcelain tile slurries since finer particle size/higher surface area is required. In order to improve grinding efficiency in wet milling, new milling machines such as "agitated/stirred media mills" have already been designed and employed. In this particular study, a combination of discontinuous ball mill and agitated media mill was utilized as a cost effective way of slip preparation for porcelain tile production. Experimental trials were performed under industrial conditions in a local tile company. The results were compared in terms of milling efficiency and energy requirements. Furthermore, the sintering behaviour and its effects on technological properties of the final product were further investigated. According to the results, such a combination was proved to generate up to 40 % energy saving for milling of porcelain tile body slip compared to that of classical ball milling.

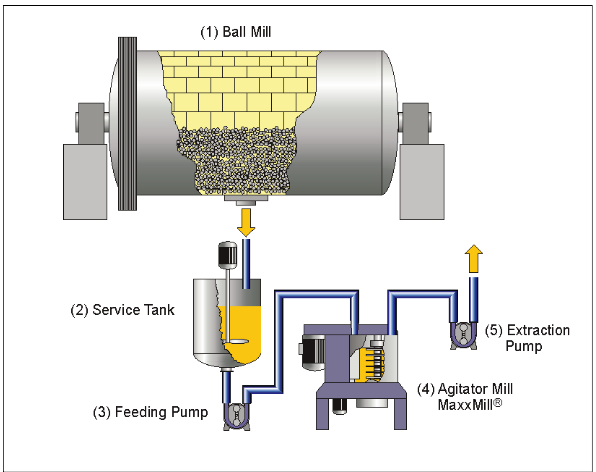

Fig. 1 Representative illustration of ball mill + agitator mill system [6]

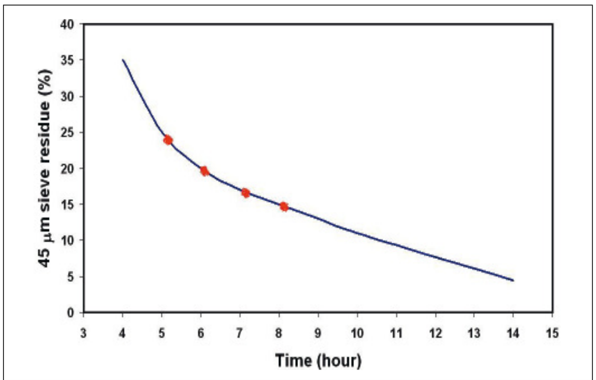

Fig. 2 Particle size reduction of the porcelain tile slip as a function of grinding time in the discontinuous ball mill

Introduction

The wet grinding process for the preparation of ceramic body slurries is widely employed during tile production [1,2]. During grinding process, control of particle size distribution is critical for successful production of ceramic tiles. The degree of grinding influences the reactivity of the components being fired, and the degree to which new phases (crystalline and glassy) are formed. Considerable reactivity favours the formation of compounds such as mullite and anorthite and helps improving the mechanical properties of the fired material. Especially in porcelain tile production, finer particle size distribution is required in order to achieve improved technological properties of the final product [3,4]. In a ball mill, grinding occurs when a particle is crushed between two pieces of grinding media. Thus, fine grinding is only possible after a relatively long grinding time due to the limit on the size of grinding media in the mill. Therefore, the efficiency of the machinery consequently falls and the cost of energy rises [3–7]. Electricity represents up to 30 % of the manufacturing cost in ceramic processing and most of the energy is consumed during the grinding operation [8].
In order to overcome the limits inherent in a ball mill system, particularly for fine grinding, the agitator ball mill has been developed, which represents a leap to a different mill type altogether. Agitator ball mills use one or several fast turning agitator shafts for introducing energy into the grinding media. By introducing energy via the agitator shafts, considerably higher volume–specific power inputs can be achieved so that smaller grinding balls can be used [4–6]. Ceramic tile production has grown in considerable amounts in recent years. Among many producing countries, Turkey is at sixth place in the world and third place in Europe with 24 ceramic tile producing companies with a capacity of approximately 350 million m² [9]. Turkish tile sector continues to expand while certain amount of efforts is directed to reduce energy consumption of the tile production in order to keep up with the fierce competition in the world market. In this study, a combination of discontinuous ball mill and agitated media mill was utilized as a cost effective way of slip preparation for porcelain tile production. The trials were carried out at *Termal Seramik San. & Tic. A. S.*, a ceramic tile company located in Bilecik, TURKEY. The agitated media mill, MaxxMill MM3, was supplied by Maschinenfabrik *Gustav Eirich GmbH & Co. KG.* Hardheim, GERMANY. The energy savings of such combination obtained were quantified and other benefits such as improved sintering behaviour and technological properties of the final product, namely breaking strength, were also discussed.

Materials and Methods

At Termal Seramik, glazed porcelain tile mixes are generally ground for 14 hours to a grinding residue of less than 5,4 % over 45 µm sieve. The specific energy consumption of this process is around 30 kWh per ton slip by using a 34 000 l capacity discontinues ball mill (with silica grinding media). As mentioned above, the agitator mill used in this study was a MaxxMill MM3 with a grinding chamber volume of 190 l. The grinding chamber of the mill is open at the top and up to 90 % of its volume is filled with 350 kg grinding media, which is made of 92 % aluminium oxide of diameter 4 to

5 mm. The grinding trials were carried out with commercial porcelain stoneware mixes, using one of the discontinuous mills combined with the agitator mill, as seen in Fig. 1. The system is constituted by ball mill (1) for pre-grinding for selected times, a service tank (2) for holding the slip, a simple pump (3) to feed the slip into the agitator mill (4) and another peristaltic extraction pump (5) to unload the ground material. The slip discharged from the ball mill was first loaded into a tank equipped with an agitator and then fed into the MaxxMill. Feeding and discharging of the slip were performed continuously via controllable peristaltic pumps. The grinding trials were carried out in two parts: in the first part, the MaxxMill was fed with the slurry, pre-ground by the discontinuous ball mill for 5, 6, 7 and 8 hours (Fig. 2), and then the agitator was operated with 8,1 m/s rotating speed and at various production rates (1,6, 2 and 2,6 t/h slip). Energy consumption and sieve residue of obtained slip were measured for each trial.

In the second part of the study, two further grinding trials were performed with the slips pre-ground in the discontinuous mill for 5,5 and 6 h. Then, the slurries were fed into the MaxxMill operated with 8,1 m/s rotating speed and at a production rate of 2,6 t/h slip. Slurries exiting the MaxxMill were screened, treated to remove any iron present and then stored in the tank serving the spray drier to form granules with a moisture content of 6 %. The spray dried granules were pressed at a pressure of 330 kg/cm² in order to obtain tiles of 33 x 33 cm. After pressing, tiles were fired at 1212°C for 32 minutes (from cold to cold) in an industrial roller kiln. Particle size analysis of the obtained slips was performed using a *Malvern* laser diffraction instrument (Hydro 2000G, UK). The technological properties of the final product such as linear firing shrinkage, water absorption, bulk density and breaking strength were measured in accordance with the standard procedures. The vitrification behavior of rectangular compacts of the representative tile bodies was studied using a double-beam optical non-contact dilatometer (*MISURA*, *Expert System Solutions*, Italy). The fired bodies were also subjected to color measurements using a UV-Vis spectrophotometer (*Minolta* 3600d). Microstructural observations were performed on polished surfaces of some selected fired samples using a scanning electron microscope (*Zeiss* Supratam 50 VP) in back-scattered (BE) electron imaging modes after sputtering with a thin layer of gold-palladium alloy in order to prevent charging.

Results and Discussion

Energy consumption and sieve residue of the porcelain tile slips were measured for each trial. The results of the trials in the first part of the study are reported in Fig. 3. As can be seen from Fig. 3a linear correlation between sieve residue and specific energy consumption could be observed. With decreasing throughput rate on MaxxMill the sieve residue decreases while the energy consumption rises accordingly. With increased pre-grinding time in ball mill the final residue at constant throughput rate in MaxxMill was reduced. The lowest total energy consumption of approx. 17 kWh/t for an identical final residue on 5,4 % > 45 µm could be achieved with 6h pre-grinding time and 2,6 t/h throughput rate on MM3. Lower throughput rates on MaxxMill and/or longer pre-grinding times in ball will further reduces sieve residue but will also reduce energy savings. Based on these first tests the most promising parameters were chosen for checking energy efficiency and sintering behaviour in further production trials. The specific energy consumption of the ball mill for preparing standard body slip and ball mill + MaxxMill for preparing of 5,5 h and 6 h pre-ground body slips were measured. The data showed that pre-grinding the slip for 5,5 and 6 h in the ball mill and then further grinding the slip in the MaxxMill to the sieve residues of 6,4 % and 5,5 % over 45 µm resulted in a total energy consumption of 16,9 kWh/t and 17,9 kWh/t, respectively, as compared with 27,8 kWh/t when the ball mill alone was used to achieve a sieve residue of 5,2 % over 45 µm. These values correspond to energy savings of 40 % (for 5,5 h) and 35 % (for 6 h) for grinding of the

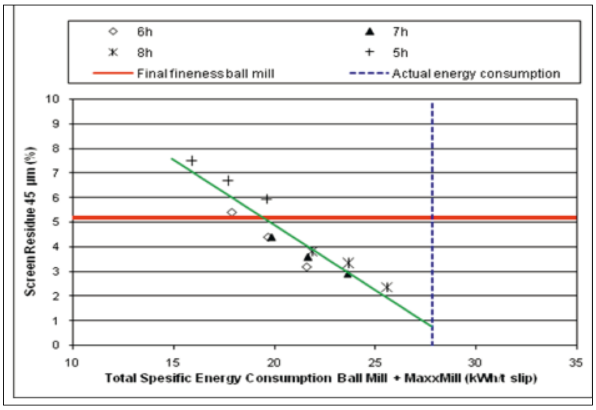

Fig. 3 Total specific energy consumption as a function of sieve residue for different pre-grinding times and throughput rates

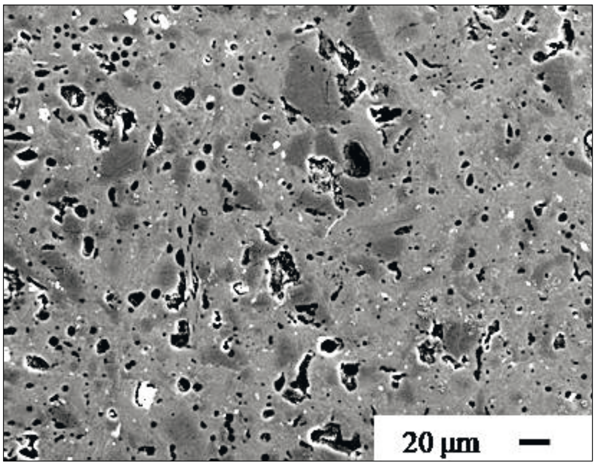

Fig. 4 A representative SEM image of the STD tile body fired at 1212°C under industrial conditions

commercial porcelain tile mix. Particle size distributions of the slurries pre-ground in the ball mill for 5,5 and 6 h and then further ground with MaxxMill (designated as 5,5 h and 6 h, respectively), are given in Tab. 1. The results were also compared with that of the standard slurry, ground in the ball mill for 14 h. According to the table, use of such a combination (discontinuous ball mill + agitated ball mill) in grinding of porcelain tile slurries achieved narrower particle size distributions than that obtained by only ball milling.

Some of the important physical properties of the ground bodies before and after fast firing are given

Tab. 1 D10, D50, D90 values and the sieve residue values of the selected slips

	D10 [µm]	D50 [µm]	D90 [µm]	Sieve Residue 45 µm [%]
Standard	1,99	12,99	51,80	5,2
5,5 h pre-grinding	1,97	12,32	49,12	6,4
6 h pre-grinding	1,87	11,69	46,71	5,5

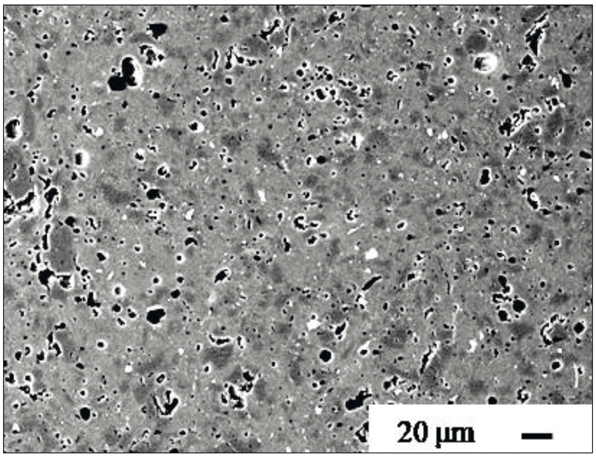

Fig. 5 A representative SEM image of 6 h pre-ground tile body fired at 1212°C under industrial conditions

Property	Standard	5,5 h pre-grinding	6 h pre-grinding
Dry Strength [N/mm^2]	1,6± 0,06	1,58± 0,13	1,62± 0,08
Firing Shrinkage [%]	7,4± 0,18	7,6± 0,03	7,9± 0,06
Water Absorption [%]	0,30± 0,02	0,22± 0,04	0,16± 0,02
Bulk Density [g/cm^3]	2,38± 0,01	2,39	2,39± 0,01
Breaking Strength [N/mm^2]	52,2± 2,34	57,3± 1,28	64,2± 2,59
L*	55,10	52,87	52,63
a*	3,42	3,57	4,12
b*	9,96	9,26	9,16

Tab. 2 Technological properties of the porcelain tile bodies fired under industrial conditions (1212°C for 32 minutes)

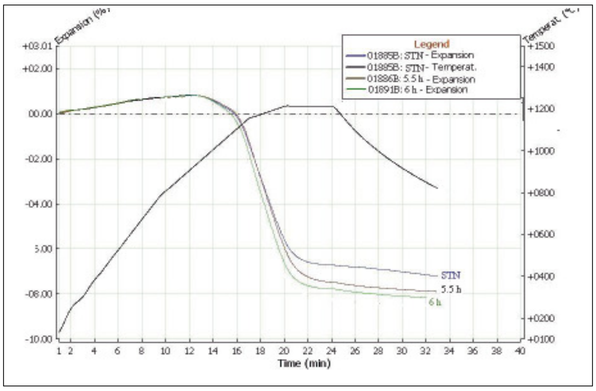

Fig. 6 Dilatometric curves of the investigated bodies (cycle: 1212°C, 32 min.)

in Tab. 2. From the results, it can be concluded that the bodies with narrower particle size distribution possess improved properties. The increase in strength is partially due to better sintering behaviour but believed to be mainly due to the diminution of negative effect of microcracks around residual quartz particles. As a very well known fact, the stress generation and associated cracking due to the presence of quartz particles tend to be severe because of the rapid displacive phase transformation of quartz and the resultant thermal expansion mismatch between quartz particles and glassy matrix on cooling. The larger quartz particles result in interconnected matrix fractures leading to poor strength [10–13]. Figs. 4 and 5 are typical back-scattered images obtained from the polished surfaces of the Std. and 6 h tile bodies both fired at 1212°C for 32 min under industrial conditions. On the images, porosity and residual quartz grains are distinguished as dark and grey areas within the structure, respectively. Finer particle size of residual quartz and smaller and rounder pores in 6 h body are clear indications of these improved technical properties in Tab. 2.

Fig. 6 presents the sintering curves of the tile bodies studied in the second part of the study. According to the curves obtained with the industrial firing cycle (1212°C, 32 min), sintering rate and final shrinkage of the investigated tile bodies increased with the decrease in particle size distribution, as expected. At the final stage of sintering where the maximum shrinkage was reached, there was no further shrinkage and bloating. In order to realize the benefits of the improved sintering kinetics, the standard body and the body pre-ground for 6 h and then further ground in the MaxxMill were fired at different firing cycles under industrial conditions and the technological properties were measured (Tab. 3). According to the results in Table 3, it was possible to satisfy the requirements of the fired porcelain tile body at faster firing regimes than usual by simply using the combination of discontinuous ball mill and agitator mill. In addition, the roller kiln capacity also increased approximately 15 % with the reduction of total firing time from 32 to 28 min.

Tab. 3
Properties of the investigated bodies fired under different industrial conditions

Property	Standard [1210°C/30']	6 h pre-grinding [1210°C/30']	Standard [1212°C/28']	6 h pre-grinding [1212°C/28']
Dry Strength [N/mm^2]	1,6 ± 0,2	1,62 ± 0,18	1,6 ± 0,2	1,62 ± 0,14
Firing Shrinkage [%]	7,3 ± 0,1	7,6	7,3 ± 0,1	7,6 ± 0,1
Water Absorption [%]	0,98 ± 0,06	0,19 ± 0,04	1,16 ± 0,02	0,26 ± 0,02
Breaking Strength [N/mm^2]	48,3 ± 2,56	58,5 ± 2,96	45,9 ± 2,12	58,1 ± 3,02

Conclusion

A combination of a discontinuous ball mill and agitator mill was employed for milling a commercial porcelain tile body under industrial conditions. It was proved that it was possible to achieve up to 40 % energy saving from classical discontinuous ball milling. Further benefits were also realized in sintering kinetics and technological properties, mainly in breaking strength.

Acknowledgement

We would like to thank Maschinenfabrik Gustav Eirich GmbH & Co. KG. for providing MaxxMill MM3 during studies.

Literature

[1] Lorici L., Contoli L.: Raw Materials Preparation: New Developments in Wet Grinding, Ceramic Acta **4** (1995) [7] 5-14
[2] Autori Vara (SALA): Raw Material Preparation and Forming of Ceramic Tiles (2002) 51–152
[3] Palacio Sergio V., Dinger Dennis R.: PSD Effects on Firing Properties of Porcelains, I-II., Am. Ceram. Soc. Bull. **75** (1996) [7] 71-83
[4] Klein G., Schulze G., Gerl S., Sachweh J.: Optimization of The MaxxMill for

Literature

[1] Lorici L., Contoli L.: Raw Materials Preparation: New Developments in Wet Grinding, Ceramic Acta **4** (1995) [7] 5-14

[2] Autori Vara (SALA): Raw Material Preparation and Forming of Ceramic Tiles (2002) 51–152

[3] Palacio Sergio V., Dinger Dennis R.: PSD Effects on Firing Properties of Porcelains, I-II., Am. Ceram. Soc. Bull. **75** (1996) [7] 71-83

[4] Klein G., Schulze G., Gerl S., Sachweh J.: Optimization of The MaxxMill for Wet Grinding of Ceramic Slips, Interceram **54** (2005) 320-327.

[5] Gerl S., Weidenhammer P.: Optimized Fine Grinding of Ceramic Slip with the Agitated Media Mill Maxxmill, Process Engineering, Ber.DKG **2** (2006) E19-E24.

[6] Nassetti G., Hessling G.: Application of the MaxxMill for the Final Grinding of Unglazed Porcelanato Tile Mixes, Process Engineering, Ber.DKG **80** (2003) 1-2 E12-E17

[7] Hogg R., Cho H.: A Review of Breakage Behavior in Fine Grinding by Stirred-Media Milling, Powder Science and Technology in Japan, No.18, (2000) 9-19

[8] Tangram Technology, Energy Efficiency in Ceramics Processing: Practical Worksheets for Industry, Energy Worksheets 1 – 8

[9] Giacomini P.: World Production and Consumption of Ceramic Tiles, Ceramic World Review, **68** (2006) 58-70.

[10] Braganc S.R., Bergmann C.P., Hubner H.: Effect of Quartz Particle Size on The Strength of Triaxial Porcelain, J. Eur. Ceram. Soc, **26** (2006) [16] 3761-3768

[11] Stathis G., Ekonomakou A., Stournaras C. J., Ftikos C.: Effect of Firing Conditions, Filler Grain Size and Quartz Content on Bending Strength and Physical Properties of Sanitaryware Porcelain, J. Eur. Ceram. Soc., **24** (2004) 2357-2366

[12] Bragan S.R.¸ Bergmann C.P.: A View of Whitewares Mechanical Strength and Microstructure, Ceramics International, **29** (2003) 801–806

[13] Carty W. M., Senapati U.: Porcelain-Raw Materials, Processing, Phase Evolution, and Mechanical Behavior, J. Am. Ceram. Soc, **81** (1998) [1] 3-20

04/09 Printed in Germany. Subject to changes.

Eirich – Individual processing technology for production and environmental protection

Eirich designs, manufactures and supplies batch and continuous machinery and systems for the processing of raw materials, compounds, waste and residues in the following fields: ceramics, glass, building materials, chemicals, molding sand, technical products, metallurgy, agricultural chemicals, food and drink, and environmental protection. Through close cooperation with our Test Centers we draw directly on the practical experience and theoretical know-how being gathered in all parts of the world. This know-how is put to innovative use in our products in the interest of greater efficiency and better environmental compatibility.

The Eirich Group worldwide:

Maschinenfabrik Gustav Eirich GmbH & Co KG
Postfach 11 60
74732 Hardheim, Germany
Phone: +49 (0) 6283 51-0
Fax: +49 (0) 6283 51-325
E-Mail: eirich@eirich.de
Internet: www.eirich.com

Groupe Eirich France SARL
Villeurbanne, France

OOO Eirich Maschinentechnik
Moscow, Russia

OOO Eirich Maschinentechnik
Dnepropetrovsk, Ukraine

Eirich Machines Inc.
Gurnee, IL, USA

Eirich Industrial Ltda.
Jandira S.P., Brazil

Nippon Eirich Co. Ltd.
Chiba, Japan

Eirich East Asia/Pacific
Seoul, Republic of Korea

Eirich Group China Ltd.
Shanghai & Beijing, P.R. China
Eirich FME
Jiangyin, Jiangsu Province, P.R. China

Eirich-Transweigh India Pvt. Ltd.
Mumbai, India

H. Birkenmayer (Pty.) Ltd.
Isando, Republic of South Africa

EIRICH