INCREASE IN VITRIFIED TILE PRODUCTION BY THE USE OF BORATE FLUX

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ABSTRACT

In this work we are reporting the use of ulexite, a borate flux, in vitrified tile production. The study describes the increase in productivity during slip making by substitution of a major quantity of feldspar by a minor amount of ulexite and clay, which helps in reducing grinding time; the increase in green production by an increase of the pressing cycle due to a substantial increase in dry MOR by the use of ulexite; and the increase in kiln productivity by reducing the kiln temperature and making the kiln cycle faster. We have also addressed the flocculation issue arising during addition of the borates in ceramic tile slip making.

1. INTRODUCTION

The rising costs of fuel and raw materials are putting great pressure on tile manufacturers around the world, particularly in the organized sector, to cut down on the production costs by substituting the currently used raw materials in the ceramic tiles industry with that of cheaper substitutes without compromising on the quality front. Many developmental efforts are also being carried out to increase productivity at various stages of ceramic tile production, which would automatically decrease the per sqm cost of tile production. Vitrified tiles are currently the highest value-added product available in any manufacturer's portfolio but considering its physical and mechanical properties, its per sqm cost of production is also the highest.

In the current work, our objective was to increase the productivity at various stages by the use of a flux called ulexite ($Na_2O.2CaO.5B_2O_3.nH_2O$), qwhich has been used by the glass industry for quite some time but has not been used in the ceramic tile industry due to one of its inherent disadvantages. The **novelty** of our work lies in the fact that we have used ulexite for vitrified tile making, resolving one of the major problems inherent to the use of ulexite or other borates, which is their tendency to cause flocculation due to the presence of Ca^{++} and Mg^{++}. In the past, people have either ignored this issue or have used other expensive borates such as Boric acid, which does not have any of flocculation-causing cations. But the use of boric acid would increase the cost of production and hence is not production friendly.

The use of borates such as Neobor (Sodium Borate), Optibor TG (Boric Acid), Vitribor (Calcium Borate), Hydroboracite (Calcium Magnesium Borate) and Ulexite (sodium calcium borate) has been referred in the literature in the past[1, 2, 3] for vitrified tile production. Several authors have also tried using borax waste and tincal for making it cost effective. Some of the common observations of these authors were that adding borate-based minerals in small quantities increased the vitrification and thereby provided better technological features. When borax waste was added to the vitrified tile, it decreased the water absorption and porosity and increased the fired strength. But the flocculation issue was not addressed in these works. Directly adding boric acid has also been the subject of many works and, unlike all other borates, it doesn't cause any flocculation issue but showed all the inherent advantages of adding borates, such as increased dry modulus of rupture, increased fired strength, reduced WA, etc. But the main problem is the high cost of boric acid and that is why borates still have not find much place in vitrified tile production[4, 5, 6].

One of the popular ways of giving dry strength to the tile body is the usage of organic binders. The development of chemical bonds between the polymer chain and the particle surface develops a three-dimensional structure which increases the mechanical strength of the body in proportion to the amount of the organic binder. But use of an organic binder has many disadvantages, which are quite well known, such as the black coring issue in the body and also the kiln dirt problem in the body, resulting in loss of quality during production. Also, one cannot store the slurry with an organic binder for a long time since organic binders are often decomposed by bacteria and an anti-bacterial agent needs to be added to stabilize slips. An inorganic binder does not have these disadvantages and can be effectively used. Since ulexite, as well as other borates, besides acting as an excellent flux also acts as an excellent binder (0.5% ulexite can increase the dry MOR by 40-60%) in the green stage, it can form a great substitute for organic binders. There have also been reports of the use of another inorganic binder[7], but the main advantage of using ulexite is that it also acts as a great flux.

Thus, the following advantages of using ulexite can be listed, which can be exploited towards cost effective production of vitrified tiles:

a) Reducing the body formulation cost by replacing a substantial amount of feldspar (6-10% depending on the firing condition and the clays used). Since ulexite is an excellent flux, we can also replace a good percentage of higher cost clays with lower cost clays and reduce the quantity of feldspar.

b) We can also increase the final slip residue after ball milling, reducing the grinding time.

c) We can utilize the excellent dry Modulus of Rupture (MOR) by use of ulexite in two ways: either reducing the tile thickness or reducing the pressing pressure resulting in an increase in the press cycle, thereby increasing the green production.

d) Due to the excellent fluxing action of ulexite, we can either reduce the peak firing temperature by 10-20 oC or make the firing cycle faster, resulting in greater kiln throughput.

2. OBJECTIVE AND SCOPE

The first and foremost objective of this work was to address the flocculation issue arising due to the use of borates, particularly ulexite, since it is the cheapest available borate, and make it production friendly in vitrified tile production.

The other objectives were to exploit the advantages of using ulexite by increasing the productivity at various stages of vitrified tile production, such as reducing the ball mill grinding time by reducing the feldspar content, increasing the press cycle due to the excellent dry MOR, and reducing the peak firing temperature or making the kiln cycle faster.

3. EXPERIMENTAL

To optimize the flow in the pilot trials, combinations of different deflocculants were tried, ranging from phosphates, phosphonates, huminates, and polycarboxylic acids to polycarboxylic acid salts and silicates. We were interested in deflocculants which utilize several deflocculation mechanisms, including cation exchange, steric repulsion and complexing of ions. We started with 0.1% of each deflocculant and then gradually increased the percentage of each deflocculant. By doing this we wanted to optimize the concentration of deflocculant level so that maximum zeta potential can be achieved and no over-deflocculation takes place[8]. Jar mills were used for lab trials with 550 g of 92% alumina balls and 1000 g of recipe. The water used for making the slip in the lab scale trials was taken from our effluent treatment plant and recycled water, which is used for industrial production. Typical TDS of such water is 1500-2500 ppm. Grinding time was fixed to get similar residue for each of the trials. The running body without ulexite is referenced R and the trial body with ulexite is referenced TU. The slip from the lab trial for both the R and the TU composition was then dried in an oven at 120 ºC for about 3 hours to achieve a final moisture of about 0.5%, and sample tiles were then made in the lab press.

The above trials were conducted using response surface design of experiments (DoE). A mathematical relation between input and output parameters was established by analysis of the design. For realizing the degree of effect of a single input on the output, the above-mentioned mathematical relation was used. A single input was varied while the others were kept constant. This analysis helped us find the optimum combination of inputs for the desired output. In the case of output flow (Y1), the inputs were deflocculants: X1, X2, X3, X4 & ulexite: X5.

After optimizing the flow of slip for the vitrified body with ulexite (TU) by DoE, we conducted two sets of production trials. For the first set of production trials, we have fixed the slip residue in both the running composition and trial compositions with similar pressing pressure to see the effect on dry MOR, fired MOR and shrinkage, keeping all the other parameters the same. And for the next set of production trials, we have increased the slip residue, considering the advantage we were getting in terms of body shrinkage, and varied the pressing pressure. We also made the kiln cycle faster considering the shrinkage advantage. A detailed discussion will follow in the next section.

Raw Material	R	TU
Clays	35-45%	30-45%
Feldspar	50-60%	45-55%
Quartz	0-2%	0-2%
Ulexite	0%	0.40-0.50%
Deflocculants	0.7-1%	0.7-1%
Binder	0.4-0.6%	0%

Table 1: Composition of running body (R) and trial body with ulexite (TU).

4. RESULTS AND DISCUSSION

The mathematical relation obtained by DoE of flow as output and concentrations of deflocculants and ulexite as input can be seen below:

$$
\begin{aligned}
Flow\ (Y1) = {} & 55.32 - (90.17 \times X1) - (85.72 \times X2) - (81.84 * X3) - (33.47 \times X4) + (52.97 \times X5) \\
& + (90.56 \times X1 \times X1) - (80.56 \times X2 \times X2) + (153.58 \times X3 \times X3) + (90.56 \times X4 \times X4) \\
& - (28.71 \times X5 \times X5) + (85.62 \times X1 \times X2) + (32.08 \times X1 \times X3) - (20.63 \times X1 \times X4) \\
& - (15.25 \times X1 \times X5) + (27.08 \times X2 \times X3) - (30.62 \times X2 \times X4) - (12.25 \times X2 \times X5) \\
& + (51.25 \times X3 \times X4) - (2.17 \times X3 \times X5) - (18.75 \times X4 \times X5)
\end{aligned}
$$

From this relation we found the effect of each input on flow. This effect is shown in the plots below.

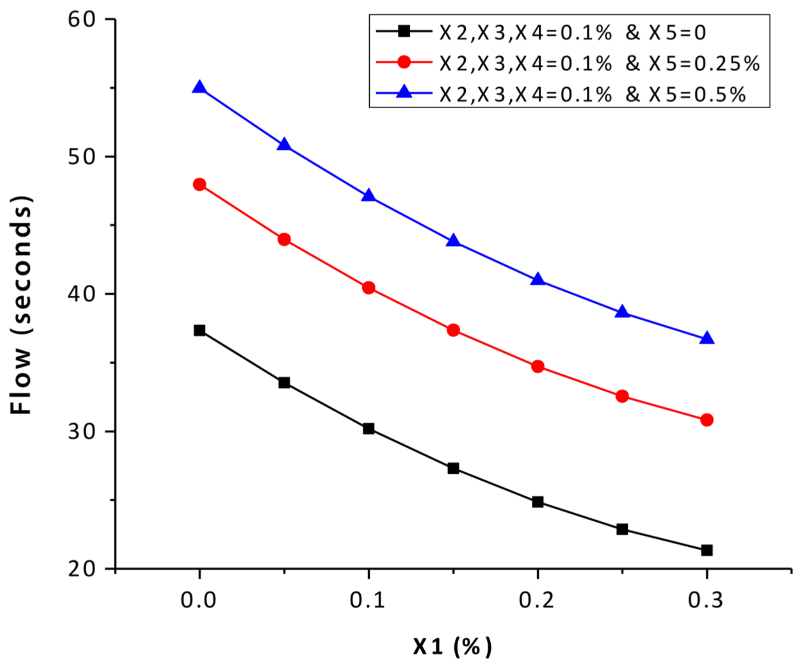

Fig. 1: Variation of flow with deflocculant X1.

Fig. 1 shows that the flow decreases with the increase in the concentration of X1 keeping all the other deflocculants fixed and varying the ulexite content. Similar results can be seen in the case of deflocculant X2 in Fig. 2. Ulexite has the tendency of decreasing the flow in both Fig. 1 and 2.

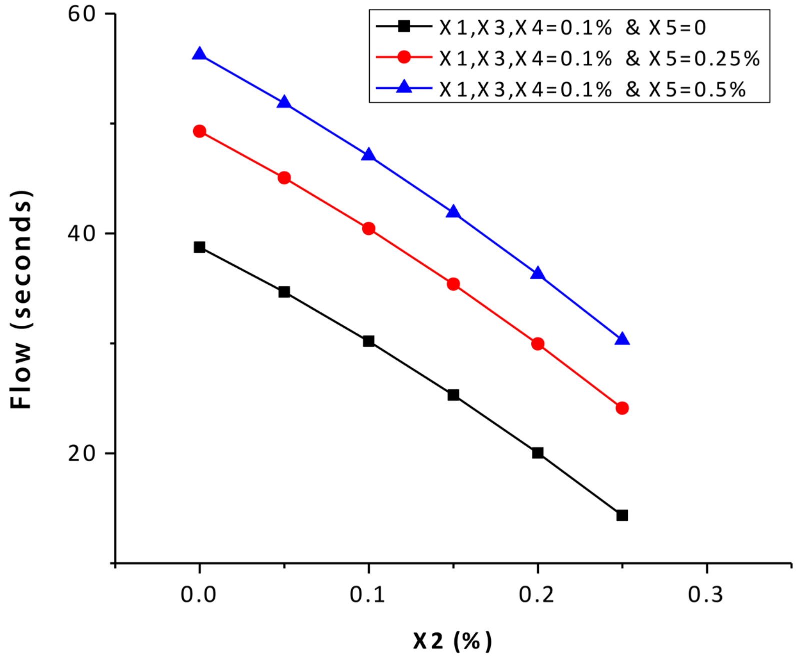

Fig. 2: Variation of flow with deflocculant X2.

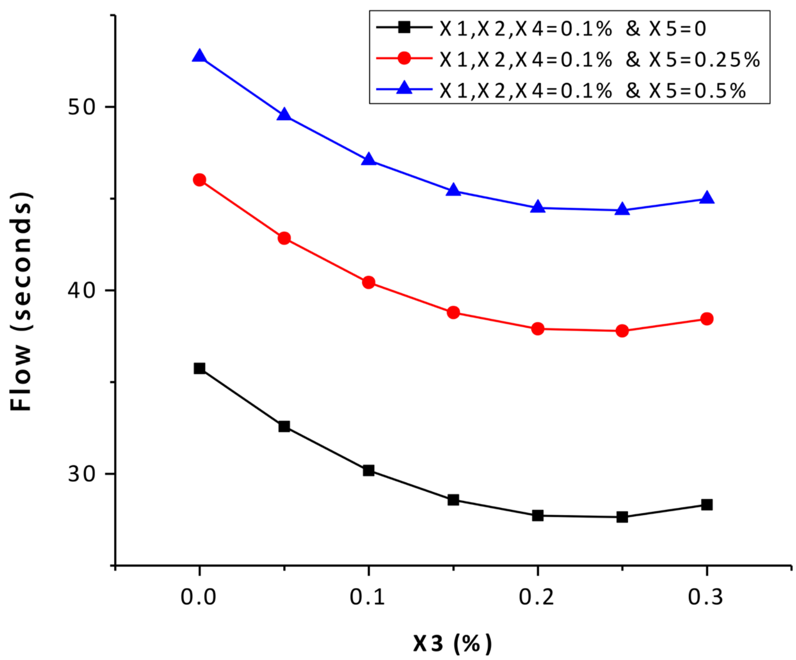

Fig. 3: Variation of flow with deflocculant X3.

The nature of plot changes in the case of Fig. 3. In this case after 0.2%, deflocculant X3 shows the tendency of flocculation and hence it should not be used beyond 0.2%. Similar is the case for deflocculant X4 shown in Fig. 4.

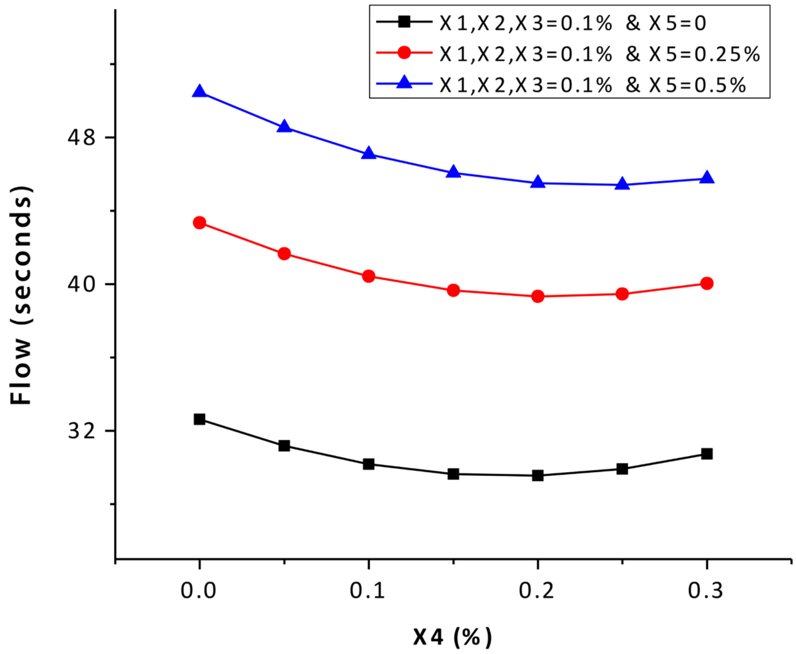

Fig. 4: Variation of flow with deflocculant X4.

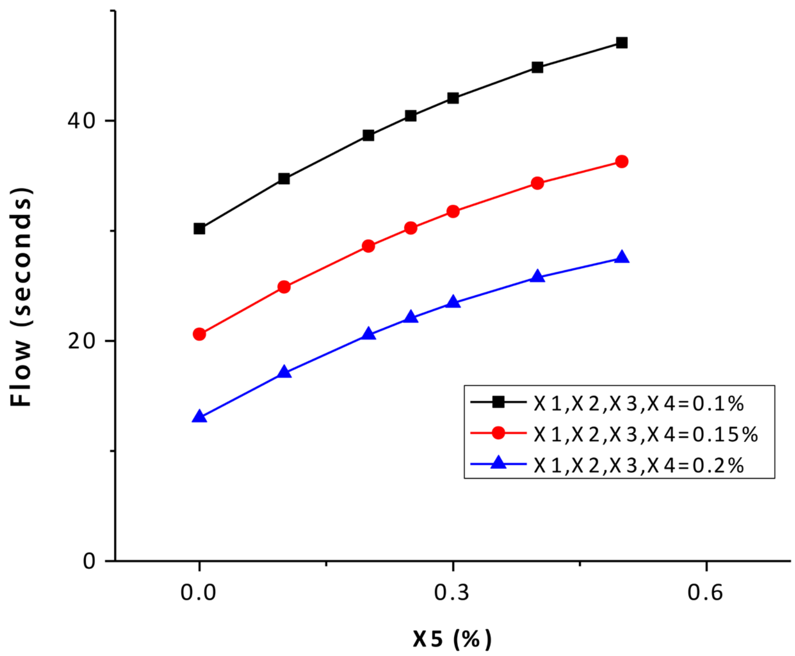

Fig. 5: Variation of flow with ulexite X5.

Figure 5 clearly shows the adverse effect of ulexite and its increasing tendency to flocculate with the increase in its concentration. But as shown in Figs. 1-4, a suitable combination of deflocculants can address this issue suitably.

From the lab trials conducted on the basis of DoE as discussed above, we finalized the most cost-effective combination of deflocculants for trial body TU, which gave us the best and consistent flow of (30-40 sec in the ford cup 4 mm diameter) with a density in the range of 1.68 to 1.72 g/cc and residue of around 4-5% on 63 micron mesh (similar to the range using running R composition). A patent application is going to be filed soon of the results of these experiments. Due to proprietary reasons we have not shown all the results of our flow trials in this paper.

Results of physical tests done in the lab are mentioned in Table 2. All the results are the average of testing of five sample tiles. All the tiles were pressed at a pressure of 120 bars.

	R	TU
Fired Shrinkage (%)	6.1	6.8
Dry MOR (kg/cm2)(Tested as per ISO 10545 part: 4)	17	24
Fired MOR (kg/cm2) (Tested as per ISO 10545 part: 4)	349	390
Water absorption (%) (Tested as per ISO 10545 part: 3)	2.5	1.7

Table 2: Results of testing of sample tiles of composition R and TU.

It can be seen from the Table 2 that we obtain higher shrinkage in the TU body even with less percentage of feldspar. We obtained a 40% increase in dry MOR and 10% increase in fired MOR with water absorption lower than that of the running body composition.

We took several production trials on the basis of the lab results. Slip was made as per the composition TU. The grinding time for the trial body TU for a similar residue was less by 30-40 min, which is due to the reduced feldspar content and increased clay content. Other parameters maintained were similar to the running body, slip residue was around 4-5% on 63 micron mesh and density was around 1.68 g/cc. Slip was then sprayed in a spray dryer maintaining all the standard conditions, and the parameters of the dust were similar to the running body. This dust was then pressed at a starting pressure of 280 bars with a press cycle of approximately 7.8 per minute. Under the above conditions, the following results were obtained for the trial body as compared to the running body.

Properties	R	TU
Dust moisture (%)	5.5	5.8
Pressure (bar)	280	280
Press cycle	7.8	7.8
Dry MOR (kg/cm^2)	16	23
Fired MOR (kg/cm^2)	530	590
Shrinkage (%)	8	8.6
W.A. (%)	0.16	0.10
Fired curvature (mm)(HOT) (Tested as per ISO 10545: part 2)	40 to 85	40 to 80
Fired curvature (mm)(After 48 hours)	20 to 50	00 to 50

Table 3: Results of the production trial with body TU as compared to body R.

It can be seen from Table 3 that with same pressing pressure and similar grain size distribution of the dust, there is a substantial increase of dry MOR by more than 40%. The firing cycle and peak temperature of body R and TU were also the same. Shrinkage of trial body TU was also higher than the running body R, while the water absorption value decreased.

Considering the advantage we were getting in terms of shrinkage, we conducted another set of trials in which we increased the slip residue from 4-5% to 4.5-5.5% on a 63 µm sieve. We got a total reduction of 1 hour in the grinding time as compared to the running body. We changed the pressing pressure from 280 to 220 bar at an interval of 20 bar and also reduced the kiln cycle time by 2 min. The results are shown below in Table 4.

Properties	R	TU a 280 bar	TU a 260 bar	TU a 240 bar	TU a 220 bar
Dust moisture (%)	5.3	5.7	5.7	5.7	5.7
Pressure (bar)	280	280	260	240	220
Press cycle	7.8	7.8	7.8	7.9	8.0
Dry MOR (kg/cm²)	17	25	21	20	18
Fired MOR (kg/cm²)	545	610	589	543	525
Shrinkage (%)	8.1	8.5	8.5	8.3	8.0
W.A. (%)	0.16	0.10	0.11	0.13	0.18
Fired curvature (mm)(HOT) (Tested as per ISO 10545: part 2)	0.3 to 0.5	0.35 to 0.70	0.4 to 0.6	0.30 to 0.55	0.25 to 0.65
Fired curvature (mm) (After 48 hours)	0.15 to 0.30	0.00 to 0.10	-0.05 to 0.20	0.05 to 0.15	0.00 to 0.20

Table 4: Results of production with body TU as compared to body R at different pressing pressure and reduced kiln cycle time by 2 min.

It can be seen from Table 4 that with the decrease of pressing pressure, the dry MOR decreased but is still higher than the running body at 280 bar. The fired MOR decreased slightly but is still well within the acceptable range. Shrinkage of the body at 220 bar pressure is slightly less than that of the running composition at 280 bar but is still within the acceptable limits. But considering the faster kiln production by 2 min and the acceptable shrinkage, fired MOR and WA, we are still at an advantage. All the other parameters were within norms.

We repeated the production trial with the TU body at 220 bar pressure several times and obtained repeatable results.

5. CONCLUSIONS

Using ulexite we obtained increased productivity at different stages of production. We obtained saving in grinding time by one hour by increasing the slip residue and change in body composition, increased press production owing to reduction of pressing pressure and increase in press cycle utilizing the considerable increase in dry MOR. We also increased kiln throughput by reducing the kiln cycle by 2 min without any considerable change in final properties of the tile.

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