

**REPORT**  
**DRY PROCESS OPTIMISATION**  
**WITH BIOKERAM**





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# INTRODUCTION

Dry granulation milling of the raw materials for tile production has increased significantly over the last number of years. The dry process consumes significantly less water and energy compared to traditional wet processing resulting in significantly lower production cost.

The quality of the final tile largely depends on the quality of the granules produced during the milling. The process requires monitoring to assure a narrow particle size distribution and homogeneous granules. The use of additives has been studied as a cost-efficient strategy to optimise granulation. Correct use of additives allow for cost saving moisture reduction in achieving optimal particle size distribution and optimise the flowability of the small granules.

Biopolymers have been used in the ceramic industry for decades, where the lack of plastic materials in the formulations demands the use of plasticisers and binders. The biopolymer strengthens the shapes after the pressing and drying stages prior to firing thereby minimising large potential losses due to breakage. We conducted two independent case studies in external technical institutes.



# CASE STUDY 1

In this case, the use of one of our liquid biopolymers is evaluated in a basic decorative red clay tile formulation which included feldspar. This formulation requires up to 14% moisture to reach an acceptable flowability.

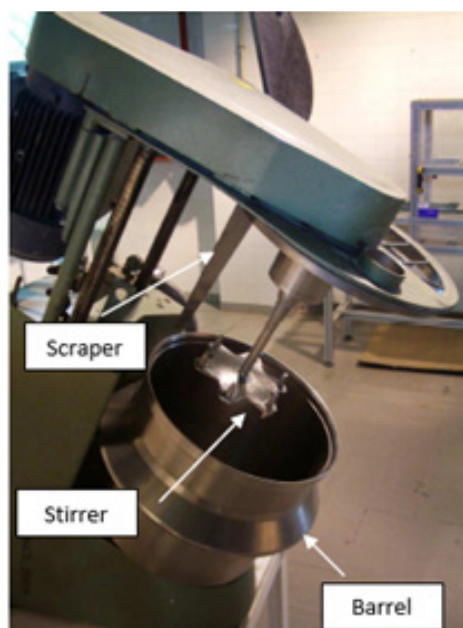
The results confirmed that agglomeration of clay particles was enhanced and the water requirement was reduced with the use of the Borregaard biopolymer.

## METHODOLOGY

The tests were carried out at the Technical Institute of Ceramics - ITC in Spain. A small Eirich M5R was used in this test, where both barrel and stirrer speeds can be modified.

The material is placed inside the barrel, the cover is lowered, the liquid is added and the first stage of nucleation occurs. Growth and breakage of the granules takes place depending on the residence time and the speed chosen at each stage of the process. It is the variation of these variables that allows forming of various sizes and shapes of granules.

To carry out the granulation, an intensive mixing of the raw materials takes place for 5 minutes at a rotor speed of 900 r.p.m. and barrel speed of 70 r.p.m. The test parameters of are described in Table 1:



Variables	Standard	Test 1	Test 2
Initial mass (g)	3000	3000	3000
Dispersion method	Direct addition	Direct addition	Direct addition
Additive (g)	-	30	30
Water (g)	354.6	319.5	267.0
Rotor speed (r.p.m.)	4800	4800	4800
Barrel speed (r.p.m.)	35	35	35
Mixing time (min)	6	6	6
Granules moisture (% on dry basis)	14.10	13.43	11.96

Table 1: Parameters of the granulation.

Granule size distribution is determined by screening with a mechanical vibrating screen, with different mesh size sieves of 125  $\mu\text{m}$ , 200  $\mu\text{m}$ , 300  $\mu\text{m}$ , 500  $\mu\text{m}$  and 750  $\mu\text{m}$  and corresponding background manifolds.

The assay was performed sieving 50 g of the samples for 7 minutes at an intermittent vibration amplitude. The tests were run twice and results were averaged.

The granule size distribution corresponding to Test 2 was not determined because the sample contained a large amount of non-granulated powder.

## RESULTS

Granule size distribution of samples Standard and Test 1 are shown in Table 2. This is represented graphically in Figure 2.

Mesh ( $\mu\text{m}$ )	Average mass (%) Standard	Average mass (%) Test 1
<125	3.5	8.3
125-200	10.9	18.3
200-300	22.9	26.6
300-500	36.5	31.0
500-750	19.6	13.2
>750	6.6	2.5

Table 2: Granule size distribution of Standard sample and Test 1

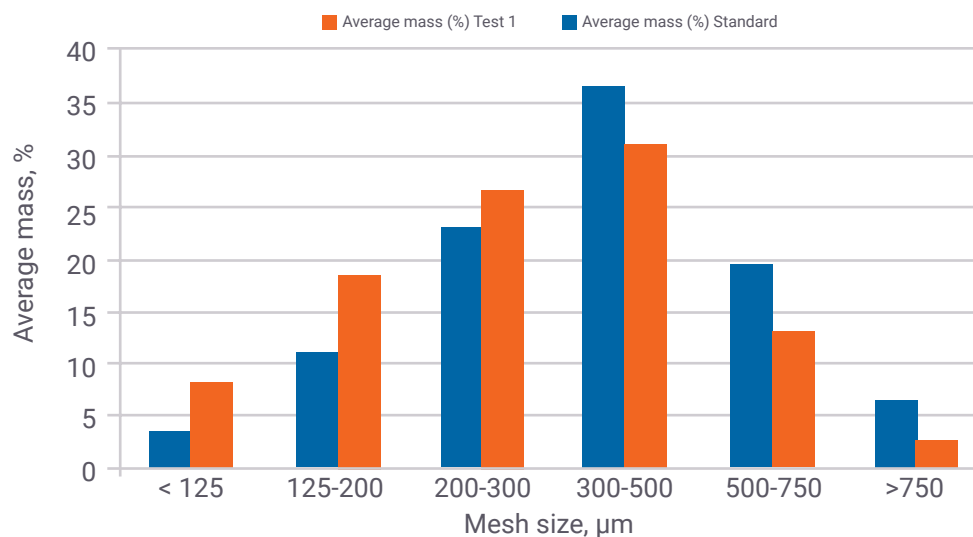


Figure 2. Granulometry distribution of the Standard sample and Test 1 Comparison.

Results show that the addition of 1% liquid additive reduces the water required by approximately 1%.

The granule moisture was reduced by a slightly lower amount (around 0.7 %) due to the contribution of water associated with the additive.

# CASE STUDY 2

A second study was performed in collaboration with Maschinenfabrik Gustav Eirich GmbH & Co KG. We used a different biopolymer than the one used in Case Study 1.

## APPARATUS

An Eirich Laboratory Mixer R01 was used in these tests:



It presents some unique capabilities, which makes it interesting for performing granulation tests:

- Rotating and inclined mixing vessel with eccentric mixing tool rotating at high speed with variable operating direction.
- Fixed wall scraper for cleaning the mixing vessel walls and to help the vertical movement of the material in the mixer.
- Interchangeable mixing tools with different geometries (star, pin and micro granulation rotor) allow the machine to be tailored perfectly to different tasks.
- Discharge: The mixing vessel can be removed from the machine frame using a quick-release coupling system.
- Technical data:
  - Capacity: 3 - 5 l / max. 8 kg
  - Mixing vessel diameter 0.23 m
  - Mixer inclination angle variable by supporting feet: 0° and 30°
  - Mixing vessel speed in two settings, driving power: max. 1.1 kW
  - Mixing tool speeds infinitely variable from 1.4 m/s to 28 m/s, driving power: up to 4 kW
  - Material temperature measurement (integrated in wall scraper)
  - Product-contacting parts stainless steel 1.4541
  - Vessel heat able for product temperatures up to 180 °C (optional)
  - Stand-Alone terminal for measurement data recording and automatic process control (optional)

## GRANULATION TESTS

For improved granulation there are some requirements:

- Fines must be bound.
- Flowability must improve.
- Surface has to be optimised.
- Water consumption must be reduced.
- Quality after firing should be maintained or improved.

As in Case Study 1, the biopolymer was used in liquid form. This is the preferred form in the European ceramic sector.

Our biopolymers may have a dual role functioning both as dispersants and binders, depending on the moisture present in the blend.

The granulation tests were run at different moisture levels and biopolymer addition rates.

After granulation, the material was aged to homogenise moisture distribution. The resultant body mix was dried in a non-heated airflow where most of the drying occurred on the surface of the granules. Further homogenisation of moisture was achieved with ageing of the dried particles. Finally, the granules were pressed at 39 bar using a standardised procedure before they were dried for 24 hours at 110 °C and fired at 1175 °C.



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*“The pressing stage shows no major differences between samples, though finishing of the surface of the probes is not equally flat. In some of the samples, roughness appears indicating either that coarser granules were most likely generated along the process, or that they are stiffer and keep their shape after the pressing stage. The firing stage shows that probes containing biopolymers shrunk less than the those without additives.”*

# RESULTS

This table describes the different moisture contents and biopolymer dosages tested in this study:

Moisture (%)	14%	14%	14%	13%	13%	13%	12%	12%	12%	14%	14%	14%	13%	13%	13%
Biopolymer (%)	0.5%	1.0%	1.5%	0.5%	1.0%	1.5%	0.5%	1.0%	1.5%	0.0%	0.1%	0.3%	0.0%	0.1%	0.3%
Clay (g)	1700	1700	1700	1700	1700	1700	1700	1700	1700	1700	1700	1700	1700	1700	1700
Feldspar (g)	800	800	800	800	800	800	800	800	800	800	800	800	800	800	800
Biopolymer (g)	12.5	25	37.5	12.5	25	37.5	12.5	37.5	62.5	0	2.5	7.5	0	2.5	7.5
Water in Biopolymer (g)	6.25	12.5	18.75	6.25	12.5	18.75	6.25	18.75	31.25	0	1.25	3.75	0	1.25	3.75
Water (g)	350	350	350	325	325	325	300	300	300	350	350	350	325	325	325
Corrected Water(g)	343.75	337.5	331.25	318.75	312.5	306.25	293.75	281.25	268.75	350	348.75	346.25	325	323.75	321.25

Humidity (%)	5.28	6.95	6.67	5.53	6.38	6.78	-	-	6.55	5.35	6.15	6.5	5	6.7	6.89
onsize (g)	2209	2137	2163	2220	2231	2306	-	-	2080	1819	2113	2048	2156	2238	2158
oversize (g)	75	218	114	51	52	51	-	-	40	282	398	407	201	256	287
Sum (g)	2284	2355	2277	2271	2283	2357	-	-	2120	2101	2511	2455	2357	2494	2445
onsize (%)	96.7	90.7	95	97.8	97.7	97.8	-	-	98.1	86.6	84.1	83.4	91.5	89.7	88.3
Bulk density (Kg/L)	1.24	1.27	1.26	1.22	1.24	1.26	-	-	1.22	1.24	1.24	1.24	1.22	1.23	1.22
Angle of repose (°)	37	35	35	45	42	45	-	-	50	34	35	35	38	39	40
Flow time 200g (s)	33	34	34	36	35	34	-	-	54	34	34	34	33	34	36

Total input (g)	2506.25	2512.5	2518.75	2506.25	2512.5	2518.75	-	-	2531.25	2500	2501.25	2503.75	2500	2501.25	2503.75
Fines(g)	222.25	157.5	241.75	235.25	229.5	161.75	-	-	411.25	399	-9.75	48.75	143	7.25	58.75
Fines (%)	8.87%	6.27%	9.60%	9.39%	9.13%	6.42%	-	-	16.25%	15.96%	-0.39%	1.95%	5.72%	0.29%	2.35%



# CONCLUSIONS

The Granular Size Distribution (GSD) graph and the table above show that for either type of mixer values are typically shifted to the left, as fines are reduced with increasing biopolymer dosage. This could be linked to the good binding properties that biopolymers typically show. This behaviour can be directly translated to better performance of the granulator, so lower amounts of raw materials need to be recycled.



# ABOUT US

Borregaard operates the world's most advanced and sustainable biorefinery. By using natural, sustainable raw materials, Borregaard produce advanced and environmentally friendly biochemicals and biomaterials that replace oil-based products. Our world-wide network of production facilities and sales offices assures the very best local service and competence where you need it. For us, providing our customers with the most dedicated technical assistance is key. Therefore, the company invests considerable resources in research and development. We continuously strive to develop wood based renewable products for new applications, and through that we contribute to delivering present alternatives to oil based synthetic products in a wide variety of industries.

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