

## Experimental evaluation of compaction load and duration effects on flowability of thermoplastic rubber (TPR) granular systems

Salvatore Coppola, Maria Francesca Pirini, and Federico Sebastiano Grasso

Elastomer Research Center, Versalis S.p.A.

### ABSTRACT

In this work, we show results obtained with two very simple experimental set-ups that allowed us predicting the ranking of flowability, at industrial scale, of a thermoplastic rubber granular system added with different flow aids.

### INTRODUCTION

It is well known that bins or silos filled with granular systems might often show severe problems with discharge of powders or granules, especially when long storage times are considered. These problems are related with (very) long time needed for flow inception and slow discharge, rat-hole and funnel flow or, in some cases, even with no flow at all. When working with industrial systems a number of important variables are to be considered. Such variables may include Particle Size Distribution (PSD), bin and outlet diameter/granule size ratios, presence of inserts close to the outlet, use of anticaking/flow improvers.

For our granular systems, some widely used set-ups for flowability testing like wedges and conical hoppers gave us results uncorrelated with the information retrieved from large-scale experiences. Other testing systems like Jenike or Schulze shear cell were instead not available at our labs. In this work, two very simple experimental setups were adopted for studying the effect of compaction load and duration on the flowability of different granular systems of industrial interest. The first set-up was used to evaluate the caking tendency of the

granular systems by measuring the normal force needed to crush a granular “cake” and is based on a parallel plate configuration mounted on a rotational rheometer. Such system needs a very limited amount of material and can be used for quick and quite reproducible testing of caking tendency. The second set-up is a lab-scale glass bin with square cross-section and a round-hole outlet at the bottom. On top of the granular system it is possible to apply a constant load by means of a chosen weight. The time needed for flow inception, as well as the total time needed for complete discharge after opening the bottom outlet, have been measured during a number of tests at varying compaction time. The results obtained instead at lab scale with the two above-mentioned setups were well correlated with those observed at larger, industrial scale.

### MATERIALS

The TPR was a SEBS industrial grade produced by Versalis SpA. The TPR granular system used in this work had a  $D_{50} = 400$  micron ca. with a maximum equivalent diameter of 1 mm and a minimum equivalent diameter of 100 micron. Two samples of silica with different mean diameter were used as flow aids, here named TL and TS, respectively. The TPR samples used for lab-scale measurements were mixed with flow aids by means of a turbo-mixer at 400 rpm for 10 min. The TPR samples used for large-scale measurements were instead mixed with flow

aids using a continuous flow- mixing device.

## METHODS

### Crushing tests

A cake of granules was prepared by compression for 30 min with a 5 N normal force in a 25 mm parallel plates geometry mounted on a MCR301 rheometer at 23°C and 50% Relative Humidity. The geometry was completed with a metal ring that plays the role of a round wall. After the compression time interval, the ring was removed and subsequently also the upper plate was lifted. The upper 25 mm plate was then changed with an upper 8 mm plate. The 8 mm plate was moved downwards to a gap of 9 mm and the test was then started with a downward velocity of 0.1 mm/s. During the test the normal force response was monitored and taken as representative of the caking tendency of the granular sample.

### Flowability tests

A glass bin having flat bottom, square cross-section (92 mm x 92 mm), and a round-hole outlet (50 mm diameter) at the bottom, was used for the lab-scale flowability tests. The sample weight used for each test was 300g. On top of the granules bed, a 2.4 Kg weight was applied. The weight was chosen in order to produce a pressure at the bottom of the glass bin of the same order of magnitude of the pressure at the bottom of the large (industrial)-scale bin. The latter was estimated by means of Janssen equation<sup>1</sup>. The large (industrial)-scale bin was geometrically similar but approximately ten times larger.

When the compaction time interval elapsed, the outlet was opened. Flow inception time was operatively defined as the time when 10% of the total mass initially present in the bin, had flowed through the outlet hole.

## RESULTS

In Fig. 1, typical results of the crushing test are shown. A striking difference is evident between the extreme cases of the TPR with no flow aid vs the TPR with TS flow aid. In such a test, the caking tendency was characterized by recording the local maximum of normal force observed during the test run. Clearly, the higher the local maximum of the normal force, the higher the caking tendency of the granular system.

This kind of test allows detecting the differences of anticaking performance of different additives. The crushing test requires a very limited amount of sample (1 g ca.) and is relatively quick and simple to be performed. Each test run was repeated three times and an average curve is shown in Fig. 1 for each sample.

From this test, we obtain a first indication that the best anticaking performance might be shown by the TS flow aid.

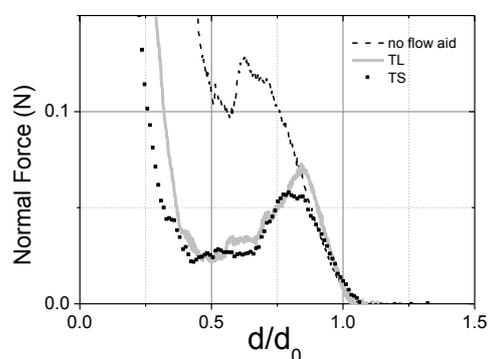


Figure 1. Crushing test: TPR granular system with TL and TS flow aid (x2 addition level) vs TPR granular system with no flow aid. The horizontal axis represents the gap normalized with the value corresponding to the first contact with the granules bed

In Fig. 2, the flowability test results are shown in terms of flow inception time as a function of the amount of flow aid added to the TPR granular system. Each sample has been tested measuring the flow inception

time after 17 h of compaction time and 69 h of compaction time. Clearly, increasing the compaction time, the flow inception is delayed. It is interesting to note that the TPR added with TS flow aid not only shows lower values with respect to the TPR added with TL flow aid but the difference is slightly enhanced with longer compaction time. The results of this test are even clearer than the crushing test results.

The observed results can be rationalized considering that the main difference between TL and TS is the mean diameter, smaller for TS. Implementing a simple geometrical model by Kojima and Elliott<sup>2</sup> we found the same qualitative trend i.e. decreasing the diameter of the flow aid may improve significantly the flowability of the whole system. This has to be done with care, however, because according to the above mentioned model a flow aid with too small diameter could even worsen the flowability.

In Fig. 3, we validated the lab-scale flowability test by comparing the results with analogous results obtained in a large, industrial-scale bin. As expected, the differences between the investigated systems were reduced at large scale. This is probably due to the fact that the compaction pressure is similar (see Methods paragraph) at lab and large scale but when the outlet is opened, at large scale the same pressure is exerted on a much larger arch. The structure is therefore weaker at larger scale and the differences due to the different flow aids are smaller.

The differences between TPR added with TL and TS flow aids are nevertheless significant, from a practical point of view, also at large (industrial)-scale. Furthermore, the observed results at industrial scale correlated very nicely with our lab-scale experimental results. This gives confidence on the concept that underlays the design of our experimental set-up. In future work, it will be interesting to use different weights for the compaction stage and for the “arch-

breaking” stage (i.e. the stage after opening the outlet).

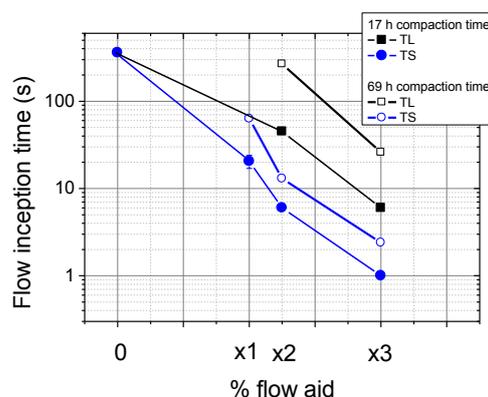


Figure 2. Flowability test. Flow inception time of lab-scale bin vs % flow aid. Three different addition levels of two different flow aids (TL and TS) have been used:  $x1 < x2 < x3$

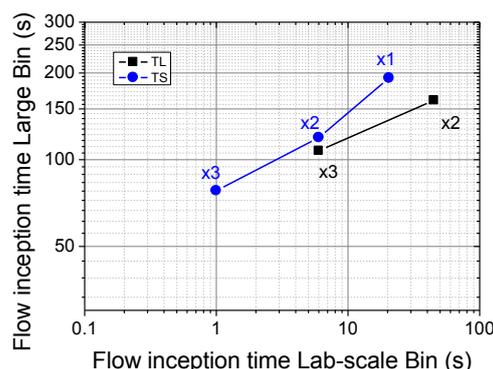


Figure 3. Flowability test. Flow inception time of large (industrial scale) bin after 144 h of compaction time vs flow inception time of lab-scale bin after 17 h of compaction time. Three different addition levels of two different flow aids (TL and TS) have been used:  $x1 < x2 < x3$

#### ACKNOWLEDGMENTS

Danilo Visani is gratefully acknowledged for his assistance with sample preparation and experimental measurements.

Versalis S.p.A., and in particular G. Regattieri, head of Versalis Elastomer

*S. Coppola et al.*

Research Center, are kindly acknowledged for the permission to publish this work.

## REFERENCES

1. Nedderman, R.M. (1992), "Statics and Kinematics of Granular Materials", Cambridge University Press, Cambridge.
2. Kojima, T. and Elliott, J.A. (2012), "A semi-empirical model relating flow properties to particle contacts in fine binary powder mixtures", *Powder Technology*, **268**, 191-202.