TALC AS ANTIBLOCKING IN LLDPE: PERFORMANCE SCREENING OF DIFFERENT TALC GRADES VERSUS OTHER MINERAL ANTIBLOCKING

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Abstract

As many plastic films tend to stick together, making difficult to separate film layers, some mineral additives are used to improve this situation. Specifically, in LLDPE films, micronized talc is often used as antiblocking agent. Thanks to the micro-roughness achievable on film surface, talc acts as a spacer between the film layers minimally affecting transparency and other mechanical properties. The presence of talc in the LLDPE film formulation interacts with other additives, creating a unique set of properties that makes talc a very effective additive for film applications.

In this paper different talc grades will be investigated for their intrinsic characteristics in comparison with other known mineral antiblocking additives to evaluate their effect in LLDPE film for final performances. A comprehensive evaluation of all the properties will be performed to rank each single tested additive for the antiblocking function, considering all the side properties including mineral additive abrasivity and bulk handling

A novel talc antiblocking additive characterized by free flowing appearance and dust-free behavior, for innovative solutions in talc handling will be also introduced.

Introduction

In order to prevent adjacent layers of polymer films from adhering to each other (blocking), antiblocking agents are normally incorporated into the polymer from which the film is made. The degree to which a film is susceptible to blocking is mainly determined by the smoothness of the surfaces; the smoother the surface, the greater is the degree of intimate physical contact and therefore the greater the blocking. The incorporation of tiny particles into the film allows to reduce the surface smoothness, minimizing the blocking. In general, inorganic minerals are used for this functions, such as: talc, diatomaceous earth, calcium carbonate, calcined clay and both ground and synthetic silica. The function of these finely divided particles is to produce asperities on the film surface and thus minimize the area of flat contact.

Antiblocking efficiency is only one of many performance criteria that must be considered in the selection of a right antiblock agent for polymer film. Other important properties are: clarity, coefficient of friction and interaction effects with processing aids. As some minerals could be quite hard, it is also important to consider the effect that those additives could have in terms of equipment wearing during their incorporation into the polymeric resin. Also additive bulk properties can play a role in the overall features to be considered when an antiblock agent has to be selected, because they might affect the economy in additive dispersion.

The purpose of this study is the evaluation of different antiblocking agents, including several talc grades, dispersed in linear low density polyethylene (LLDPE), comparing different sets of properties achievable into a blown LLDPE film.

Experimental

Materials

A general purpose M.I. 1.0 g/10min butene LLDPE resin was used for both antiblocking masterbatch granules and film production. The resin is characterized by a density of 0.918 g/cc.

Different inorganic additives were used as antiblocking agents and their properties are summarized in Table 1. All of them are from natural source with the exception of amorphous synthetic silica. In this study, six different talc grades from IMI Fabi Spa were investigated versus calcium carbonate, diatomaceous earth, calcined clay and synthetic silica. The talc grades are characterized by different particle size distributions and some of the investigated grades are also coated for better dispersion in polymer and lower interaction with processing aids. In Figures 8 to 12, a SEM image at the same magnification of the five different antiblocking additives is showed for a direct visual comparison of the inorganic products.

Abrasivity test, summarized in Table 1, consists in the amount of copper abraded by a thin copper net where a water slurry of the additive has insisted for 120 minutes under a controlled contact condition. The higher the abrasivity value, the stronger the expected wearing achievable during mineral processing.

Processing

The antiblocking additives were pre-dispersed into the LLDPE resin to form a masterbatch. All the antiblocking additives were loaded into the masterbatch to achieve a final loading of 3000 ppm into the blown film. Also process stabilizers and slip agent (Erucamide at 800 ppm final loading on the film) were added to each

Paper presented at*: 2017 SPE international Polyolefin Conference Feb 27th-Mar 1st 2017 – Houston, TX - USA*

masterbatch. Masterbatches were produced by means of a co-rotating intermeshing twin screw extruder, purging the feeding area with nitrogen to minimize oxidation. Masterbatch was precisely mixed to the resin to achieve the final loading of 3000ppm of antiblocking additive and 800 ppm of Erucamide; the blend was extruded into a laboratory blown film line, achieving a 45 micron film.

Neat LLDPE resin (without antiblocking additives and slip agent) was extruded to achieve a reference in the study. Also a sample containing slip agent (800 ppm Erucamide) has been prepared to measure the effect of slip agent without inorganic antiblock additive (sample ID is LLDPE).

Testing

The induced blocking test procedure used the parallel plate method as per ASTM D3354, to determine the degree of blocking. In preparing the samples, squared pieces of film were cut from the layflat tubing; the double layer was separated and discharged from static charges before being reunited and conditioned in recirculating forced air oven for 24h at 60°C, under controlled pressure. All samples were tested for antiblocking final loading, to confirm the exact 3000 ppm antiblock content.

The slip performance, as measured by static coefficient of friction (COF), follows the test procedure of ASTM D1894.

Optical properties were measured according to: ASTM D1003 for transparency (haze), ASTM D2244 for color (yellowness) and ASTM D2456 for surface appearance (gloss at 60°).

Mechanical properties were tested as well, according to: ASTM D882 (tensile strength at yield – machine direction) and ASTM D1709 (dart drop test).

Results and discussion

Antiblocking properties

To compare the relative antiblocking performance of the examined inorganic additives, the induced blocking at 60°C has been used, such temperature is rather selective and it emphasizes the antiblocking efficiency. In Figure 1, the results are plotted.

It appears that the modification of neat LLDPE with slip agent makes the film opening worse (better surface adhesion), while the addition of inorganic antiblock agents improves the film separation. The worse antiblock agent in this evaluation results to be the calcined clay, followed by calcium carbonate. Talc samples are in between, while the highest efficiency is recorded by both diatomaceous earth and synthetic silica samples.

Concerning the talc samples behavior, results can be split into two groups: finer grades (NB140c and NB140Tc) and coarser grades (all the other talc samples). In general, the coarser the talc size, the higher the antiblocking efficiency.

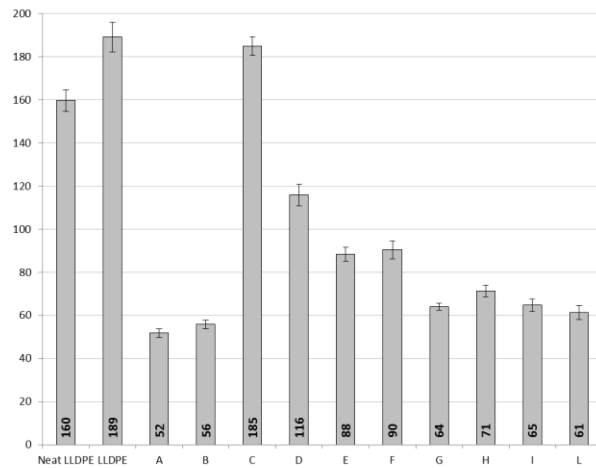

Figure 1: *Antiblocking [g] after re-blocking at 60°C of 45μm LLDPE blown film, modified with 3000ppm of different antiblocking additives and 800ppm of Erucamide - according to ASTM D3354*

Optical properties

The haze behavior in the study can be observed from Figure 2. The addition of additive masterbatch barely affects the transparency (LLDPE versus LLDPE) and the haze variation in the samples in mainly due to the inorganic additives themselves.

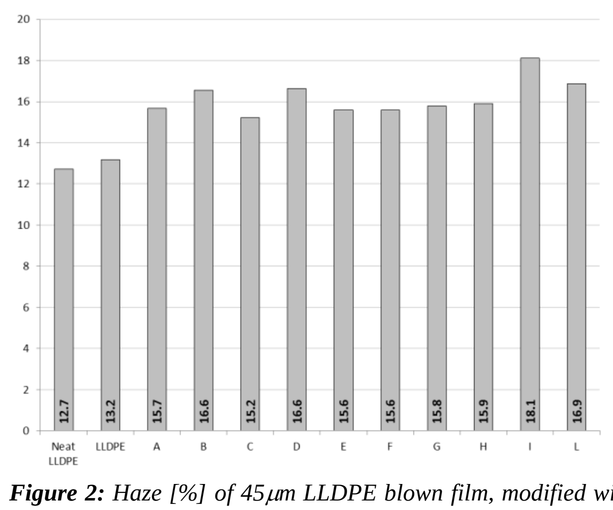

Figure 2: *Haze [%] of 45μm LLDPE blown film, modified with 3000ppm of different antiblocking additives and 800ppm of Erucamide - according to ASTM D1003*

The best behavior is recorded by calcined clay, followed by diatomaceous earth and fine talc. Synthetic silica performs as per calcium carbonate and the coarsest talc samples (CHB2 and NoBlock-S). In the case of talc samples, haze level achieved per same talc loading is directly linked with the talc particle size distribution: the finer the talc size, the lower the haze.

In terms of color, the yellow index was set as indication for both process condition and additive modification. The intermediate usage of masterbatch for blown film production, didn't affect the resin color, as visible from yellowness values recorded between neat LLDPE and LLDPE (raw resin modified with masterbatch containing slip agent only).

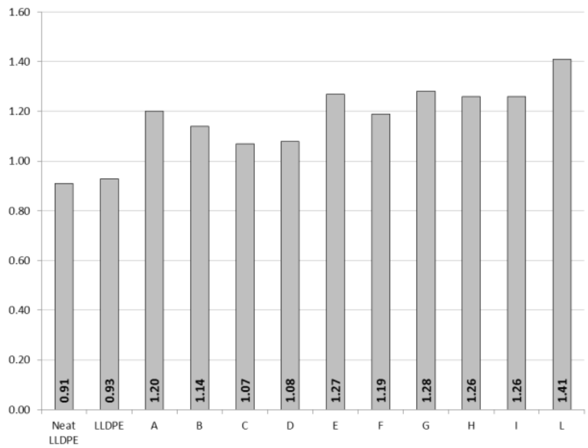

Figure 3: *Yellowness [-] of 45μm LLDPE blown film, modified with 3000ppm of different antiblocking additives and 800ppm of Erucamide - according to ASTM D2244*

The yellowness gap visible from Figure 3 data is due to the antiblocking additives. The more neutral additive are both calcined clay and calcium carbonate, while talc samples show the more visible variation.

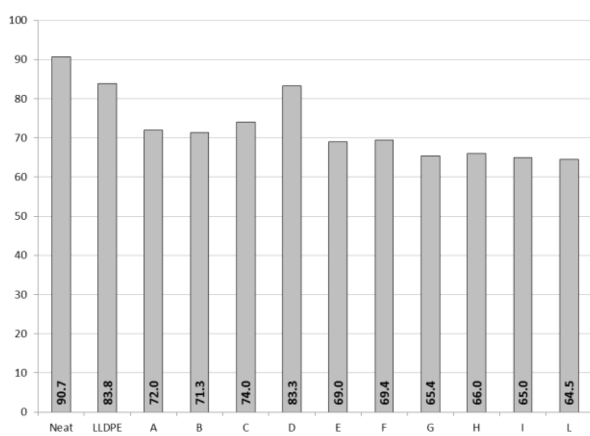

Figure 4: *Gloss at 60° [%] of 45μm LLDPE blown film, modified with 3000ppm of different antiblocking additives and 800ppm of Erucamide - according to ASTM D2244*

The effect of foreign particles into the LLDPE film is immediately visible on recorded gloss data; In fact, the original value of LLDPE gloss records a significant reduction once modified with additives. The slip agent itself contributes to the gloss reduction; differently from the other antiblock additives, calcium carbonate doesn't contribute to the gloss reduction. In Figure 4, test results are summarized.

Slip properties

Slip properties were measured as per static coefficient of friction (COF). From Figure 5 it is clearly visible the improvement achieved by adding the slip agent to neat LLDPE. The further reduction is then achieved because of the micro roughness developed by the presence of inorganic antiblocking additives. The main exception can be recorded for synthetic silica that minimally improve the COF. The strong interaction existing between synthetic silica and slip agent is clear because of the extremely high specific surface of the inorganic additive: part of the slip additive was absorbed by silica, making it no longer available on the film surface. Also fine talc shows this tendency (but in minor extent). The higher micronized grade (NB140c) shows the highest COF among the talc samples; its surface coated version (NB140Tc) records an improvement in COF because of the lower interaction with slip agent. The best result was achieved with NB240TL (coated).

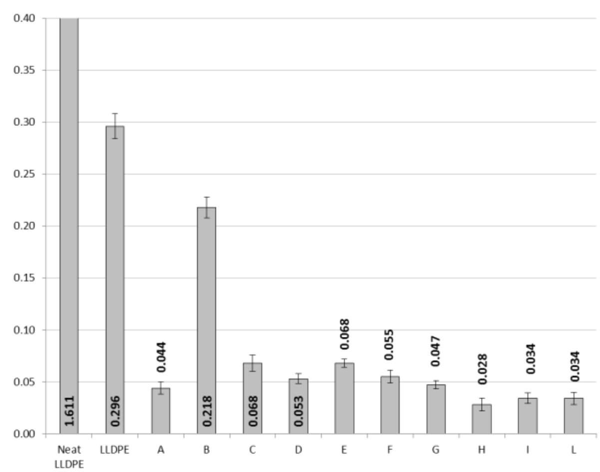

***Figure 5**: Static coefficient of friction (COF) [-] of 45μm LLDPE blown film, modified with 3000ppm of different antiblocking additives and 800ppm of Erucamide - according to ASTM D1894*

In this work, the interaction effect between inorganic antiblocking and polymer processing aids (PPA) was not investigated. PPA are used to improve the melt fracture in LLDPE during extrusions as well as to minimize the die build-up (such as fluoropolymers) The same considerations made on slip properties can be partly extended to PPA, as general behavior. It means that coated talc will exhibit lower interactions with PPAs keeping them more available for their primary function and thus allowing a certain cost saving on PPA lower loading. .

Mechanical properties

Referring to Figure 6, tensile strength (machine direction) of neat LLDPE is always negatively affected by the presence of foreign additives. The slip agent (800 ppm of Erucamide) minimally affects the tensile properties of the film, but the inorganic additives contribution is more visible. In particular, both calcined clay and calcium carbonate show the strongest effect. All the other additives are, more or less, on the same level.

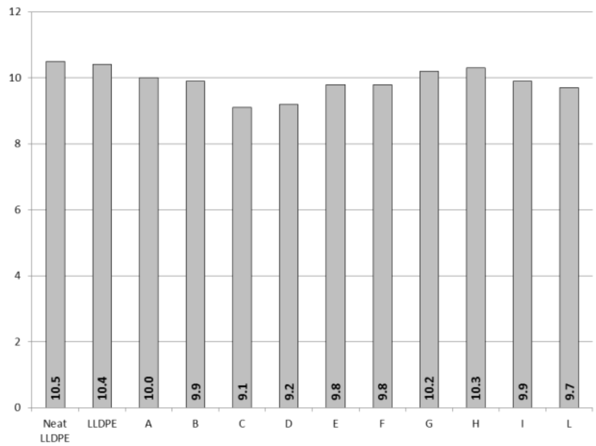

Figure 6: *Tensile strength at yield [MPa] (machine direction) of 45μm LLDPE blown film, modified with 3000ppm of different antiblocking additives and 800ppm of Erucamide - according to ASTM D882*

A similar behavior can be observed in Figure 7 for dart drop test too. Dart drop test records similar data for all the tested additives with the exception of synthetic silica that showed the worse value.

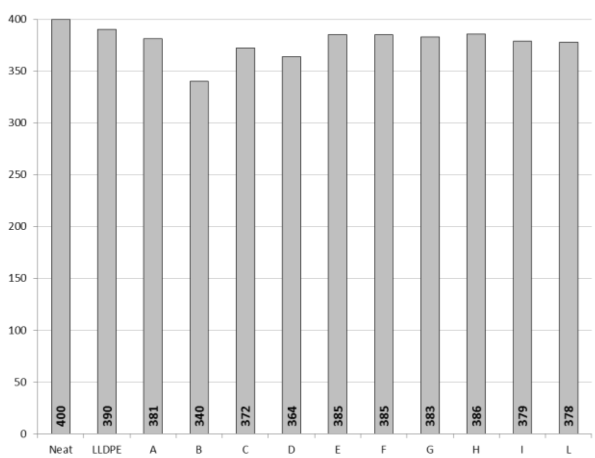

Figure 7: *Dart drop[g] of 45μm LLDPE blown film, modified with 3000ppm of different antiblocking additives and 800ppm of Erucamide - according to ASTM D882*

Conclusions

For a proper evaluation of an antiblocking additive it is necessary to consider a combination of different properties to cover all the different properties of interest for a LLDPE film.

In general, together with antiblocking evaluation, the most important properties refer to both clarity and coefficient of friction. Performing a simple scoring (Table 2), where the scores are meant as a value calculated between the worse (score = 0) and the best (score =10) in the evaluated series of experimental data, it is possible to rank the different additives under those three properties. A very simple calculation (Table 3) addresses to diatomaceous earth (Sample A) the best in class product, followed by talc NB240L and NB240TL (samples G and H).

Then, considering other properties such as the abrasivity of the different additives, the bulk density for easy handling and the price, the ranking (Table 4) changes in favor of most of the talc samples, recording talc NB240 (sample G) as the best in class. Talc samples show a more balanced set of properties including some side properties not always considered as important during an additive evaluation, but potentially critical during its utilization. In particular, the abrasivity of the inorganic additive could affect both mixing equipment and downstream apparatus used in film handling such as trimming device. The higher bulk density, available for compacted talc, allows to achieve a better handling, especially during the production of concentrates to be used in blown film production.

In this respect, the sample NoBlock-S, showing a good set of properties, is characterized by an exceptional flowability. Because of the spherical agglomerates, NoBlock-S flows in every conditions, allowing its usage even in the more severe handling conditions. Also, it develops very little dust during handling, making this additive perfect for all the environments sensitive to dust pollution.

Key Words: LLDPE, antiblocking, talc, synthetic silica, calcined clay, calcium carbonate,

Additive	ID	bulk density [g/cc]	specific surface [m^2/g]	abrasivity [mg]	fineness D_{50} [μm]	fineness D_{98} [μm]	brightness CIE-L [-]	brightness CIE-b [-]	coated	compacted
Diatomaceous earth	**A**	0.23	2.00	121	11.3	25.9	97.2	1.8	no	no
Synthetic silica	**B**	0.16	380.79	30	5.7	11.4	98.5	0.3	no	no
Calcined Clay	**C**	0.50	11.00	57	3.8	15.2	96.5	3.0	no	no
Calcium Carbonate	**D**	0.91	3.00	25	4.1	15.9	95.6	3.1	yes	no
Talc NB140c	**E**	0.90	7.62	6	6.1	15.8	97.0	0.7	no	yes
Talc NB140Tc	**F**	0.90	5.76	5	6.8	18.7	96.9	0.8	yes	yes
Talc NB 240L	**G**	0.60	6.44	7	8.3	24.1	97.5	0.5	no	yes
Talc NB 240LT	**H**	0.60	4.59	6	8.3	23.5	97.3	0.8	yes	yes
Talc NoBlock-s	**I**	0.67	8.05	6	9.2	25.6	96.7	1.6	no	yes
Talc CHB2	**L**	0.29	6.74	7	11.2	37.2	95.3	0.7	no	no

Table 1: *main properties of the different inorganic additives used as antiblocking agents in LLDPE films. Diatomaceous earth, synthetic silica, calcined clay and calcium carbonate are samples secured on the market, while all the talc samples are produced by IMI Fabi Spa.*

Property	**LLDPE**	**A**	**B**	**C**	**D**	**E**	**F**	**G**	**H**	**I**	**L**
Antiblocking	0.0	10.0	9.7	0.3	5.3	7.3	7.2	9.1	8.6	9.1	9.3
Transparency	10.0	4.9	3.1	5.9	3.0	5.1	5.1	4.7	4.5	0.0	2.5
Slip	0.0	9.4	2.9	8.5	9.1	8.5	9.0	9.3	10.0	9.8	9.8
Abrasivity	10.0	0.0	7.8	5.5	8.3	9.9	10.0	9.8	9.9	9.8	9.9
Handling	9.9	0.9	0.0	4.5	10.0	9.9	9.9	5.9	5.9	1.7	6.8
Price	10.0	8.7	0.0	9.7	10.0	9.5	9.3	9.7	9.5	9.9	9.3

Table 2: *relative scoring for different examined properties. For each one, a proportional score in the range 0 (worse) – 10 (best) is recorded among the experimental data recorded in the experiment. For transparency, haze was considered, while for handling, the powder loose bulk density was used for scoring.*

Property	Weight [%]	**LLDPE**	**A**	**B**	**C**	**D**	**E**	**F**	**G**	**H**	**I**	**L**
Antiblocking	33.34	0.00	3.33	3.24	0.10	1.78	2.45	2.40	3.04	2.86	3.02	3.10
Transparency	33.33	3.33	1.65	1.05	1.95	1.00	1.70	1.70	1.56	1.49	0.00	0.84
Slip	33.33	0.00	3.13	0.97	2.84	3.02	2.84	3.00	3.10	3.33	3.26	3.26
total	100	**3.33**	**8.11**	**5.25**	**4.89**	**5.80**	**6.98**	**7.09**	**7.70**	**7.69**	**6.28**	**7.20**

Table 3: *Scorecard calculated on the relative scores listed in Table 2 and weighed for each considered properties. In this scorecard, properties such as antiblocking, transparency (Haze) and slip (static COF) were considered at same relative weight. The higher the score, the better the set of properties.*

Property	Weight [%]	**LLDPE**	**A**	**B**	**C**	**D**	**E**	**F**	**G**	**H**	**I**	**L**
Antiblocking	25	0.00	2.50	2.43	0.07	1.33	1.84	1.80	2.28	2.14	2.26	2.33
Transparency	25	2.50	1.23	0.78	1.47	0.75	1.27	1.27	1.17	1.12	0.00	0.63
Slip	20	0.00	1.88	0.58	1.70	1.81	1.70	1.80	1.86	2.00	1.96	1.96
Abrasivity	10	1.00	0.00	0.78	0.55	0.83	0.99	1.00	0.98	0.99	0.98	0.99
Handling	10	0.99	0.09	0.00	0.45	1.00	0.99	0.99	0.59	0.59	0.17	0.68
Price	10	1.00	0.87	0.00	0.97	1.00	0.95	0.93	0.97	0.95	0.99	0.93
total	100	**5.49**	**6.58**	**4.58**	**5.22**	**6.73**	**7.73**	**7.78**	**7.85**	**7.79**	**6.36**	**7.51**

Table 4: *scorecard calculated on the relative scores listed in Table 2 and weighted for each considered properties. In this scorecard, properties such as as antiblocking, transparency (Haze), slip (static COF), Handling (additive bulk density), additive abrasivity and price were considered at different relative weight. Antiblocking, slip and transparency were set at the highest weight (70% of total), while the other properties were considered for the remaining 30%. The higher the score, the better the set of properties. .*

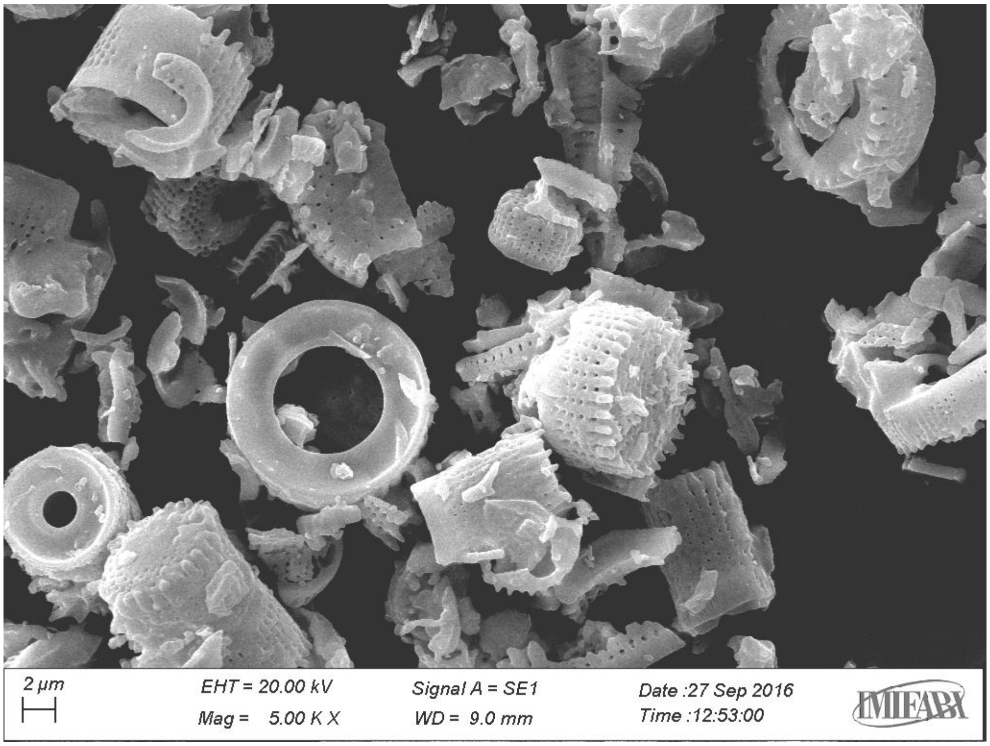

Figure 8:*SEM image of a powder sample of diatomaceous earth*

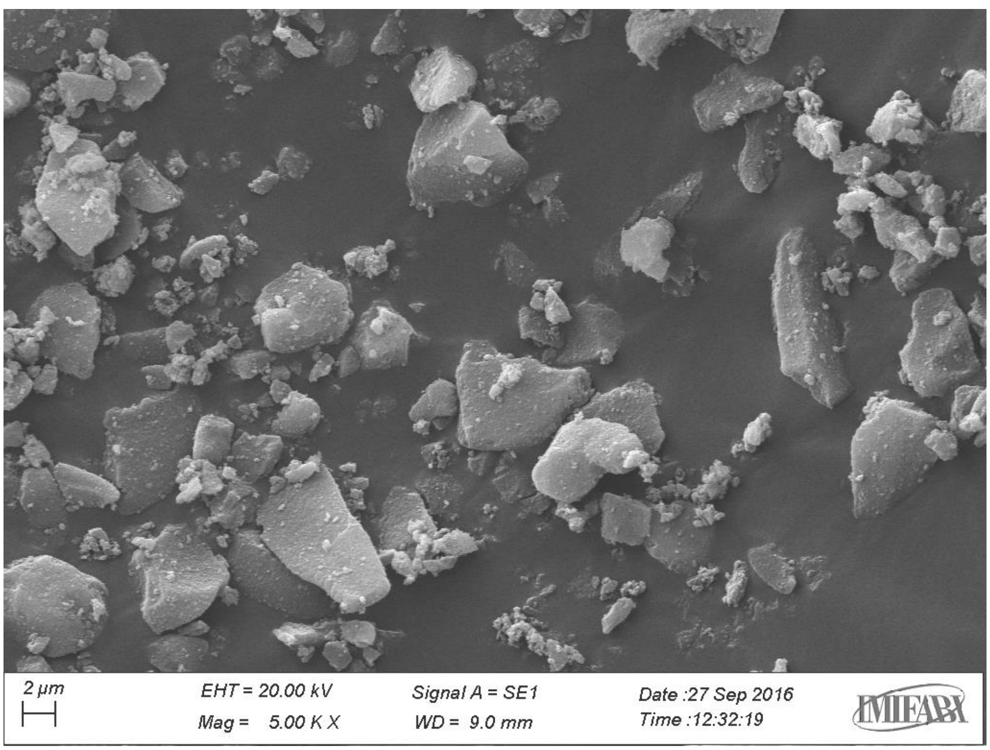

Figura 9: *SEM image of a powder sample of synthetic silica*

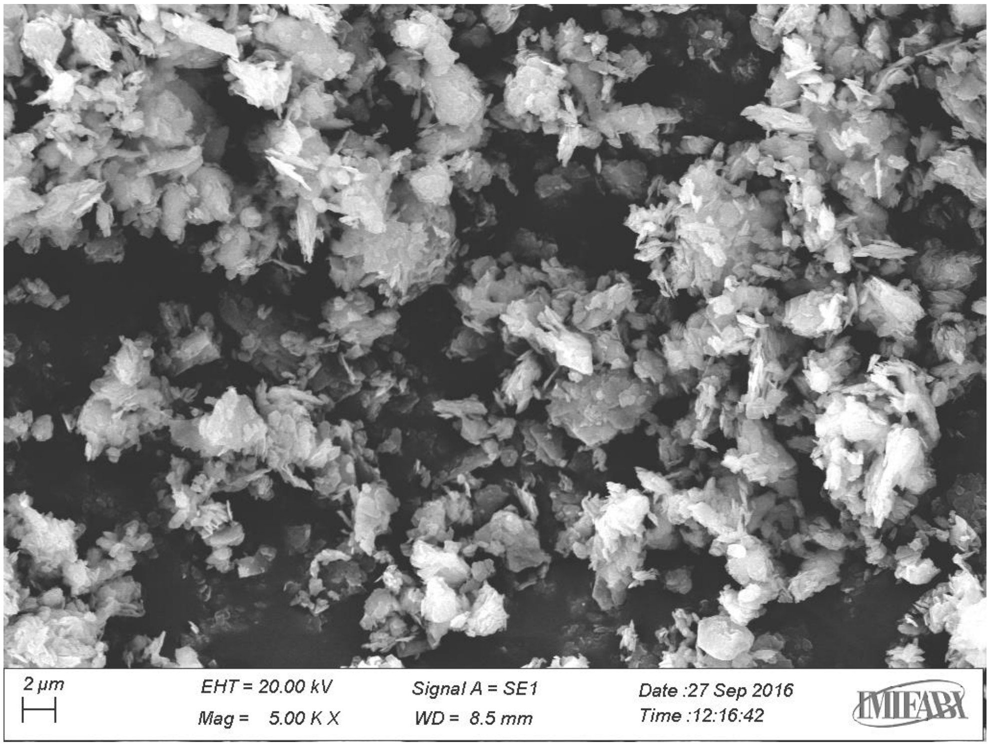

Figure 10: *SEM image of a calcined clay*

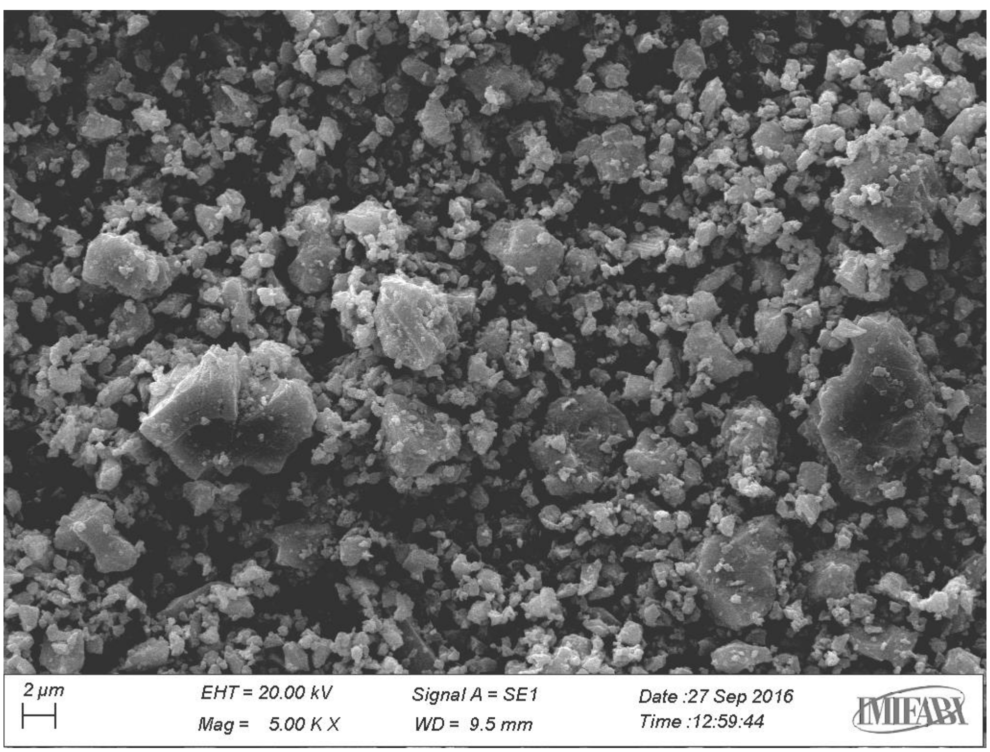

Figure 11: *SEM Image of ground calcium carbonate*

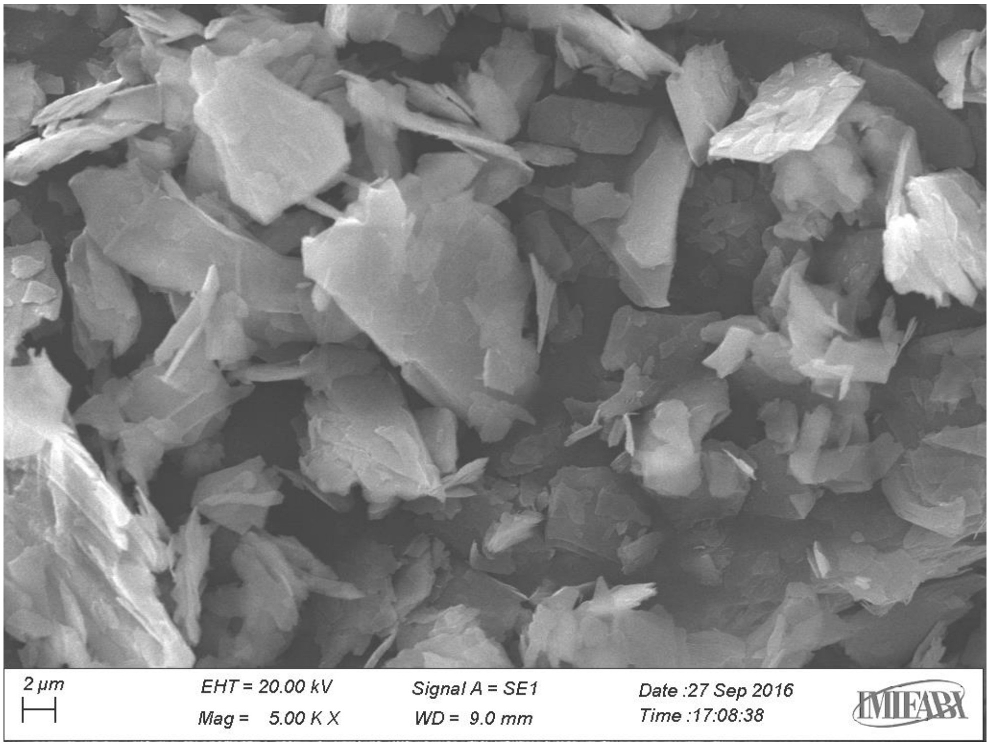

Figure 12: *SEM image of micronized platy talc*