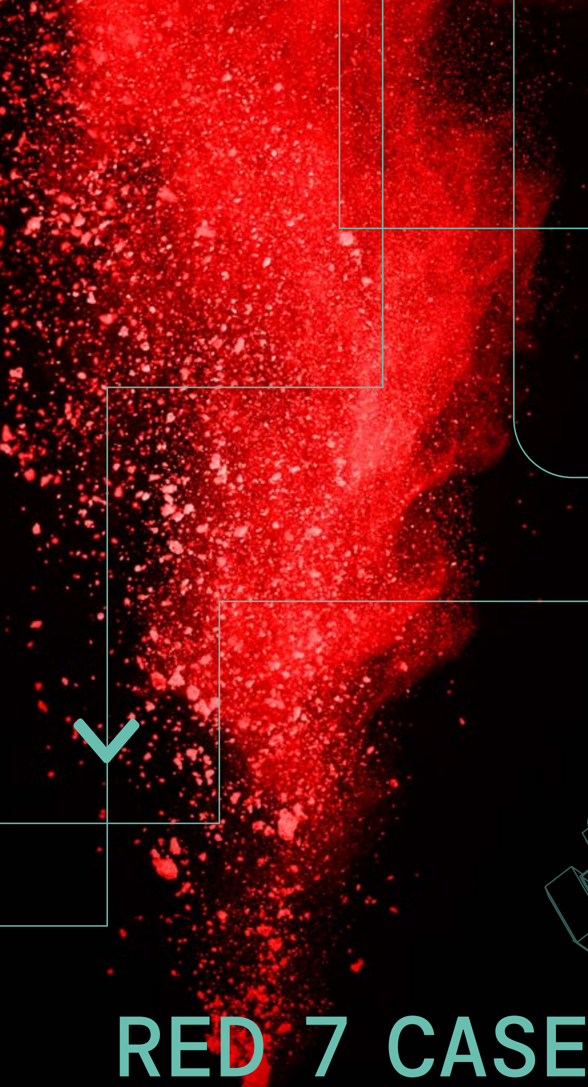

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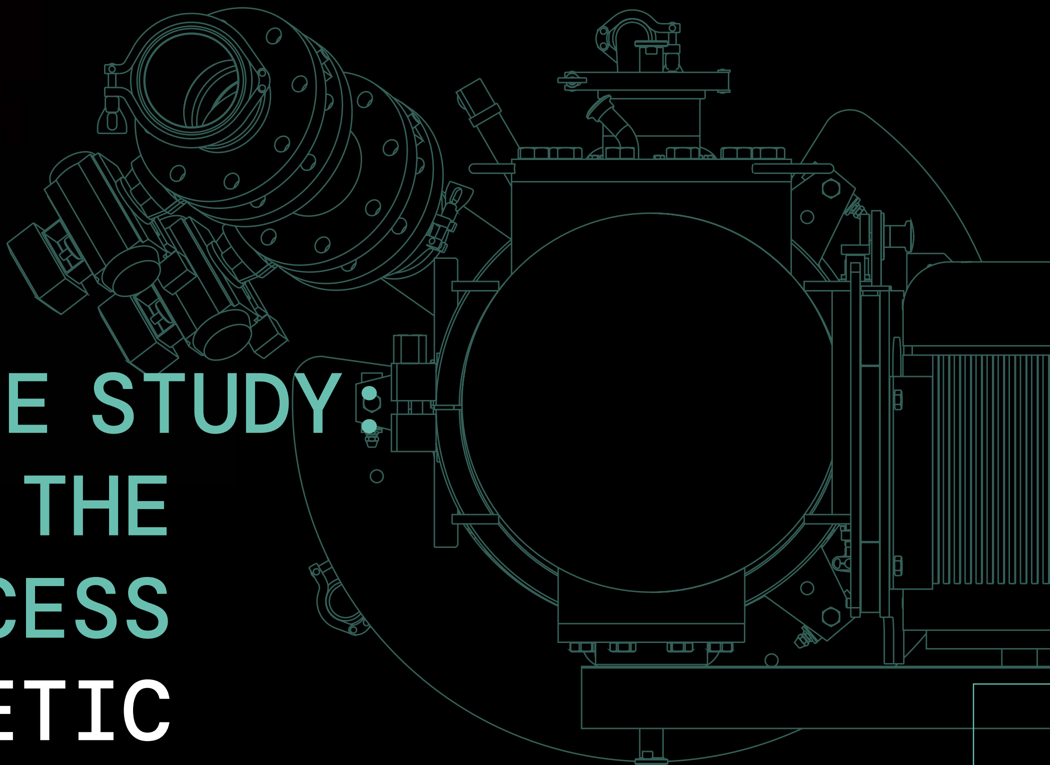

RED 7 CASE STUDY DEFINING THE BEST PROCESS FOR COSMETIC MICRONIZATION

WHITEPAPER SCHEDIO SA
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Written in cooperation with:
OMNICOS GROUP

INTRO

Particle size distribution (PSD) and uniformity are critical for cosmetic powders and the most common technique used is micronization to get the best, final product. Since PSD of powders determine the quality of products, the role of micronization is very critical. Micronization can be done either on entire formulations, or on the single cosmetic ingredients.

Micronization can offer several advantages when used on cosmetic pure pigments:

- Improved color intensity: Smaller particles scatter light more effectively, resulting in a more vibrant and intense color.
- Increased stability: Smaller particles are less likely to settle or separate, which can improve the stability of a cosmetic product containing pure pigments.
- Improved dispersion: Smaller particles are more easily dispersed in a liquid or cream, which can improve the overall performance of a product containing pure pigments.
- Increased sunscreen efficacy: Micronized titanium dioxide and zinc oxide are widely used as sunscreen ingredients, they provide better UV protection due to the smaller particle size.
- Improved aesthetic appeal: Pure pigments often provide a very bold and striking color, but can be difficult to apply due to their coarseness. Micronization can help to make these pigments more user-friendly and pleasant to use.
- Improved feel: The particle size of pure pigments is reduced through micronization, the powder becomes finer and smoother. This can result in a more pleasant sensation when the product is applied to the skin, as the powder will not feel as gritty or rough, resulting in a more natural and even appearance. Additionally, the smaller particle size can help to reduce the amount of powder needed to achieve the desired color, which can make the product feel lighter on the skin.

PROCESS CHALLENGES:

However the micronization process applied to cosmetic raw materials or formulations present multiple challenges, like cleaning, consistent particle size distributions, scalability, abrasion, contamination and clogging. Certain raw materials cause particular clogging challenges due to their physical composition, and the RED7 tested for the preparation of this white paper is not an exception.

RED7 is an organic pigment, particularly oily and commonly used in cosmetics for lipstick manufacturing. Its oily properties and poor flowability may oblige manufacturers to mix it with binders, in order to process it in mechanical mill, to obtain fine particles, while jet millings was always thought as not applicable, until today.

This case study will discuss the challenges of the current process for particle size distribution and how Schedio helped Omnicos to define a new way to help with improving the micronization process of the pure RED7.

WHAT ARE THE ADVANTAGES AND DISADVANTAGES OF THE DIFFERENT MILLS FOR COSMETIC MICRONIZATION?

Depending on both the raw product proprieties and the typology of the finished product (cream, powder, lipstick etc.), the design and engineering of ad hoc applications is a key factor. This crucial step is undervalued and limits significantly the potential of the process and equipment. There can also be disadvantages with specific mills, including spiral jet mills and mechanical mills, the two most commonly used technologies for micronization of cosmetic products.

1. The challenges that arise when using spiral jet mills for cosmetic powders:
   - Mixtures and fat powders are difficult to process with spiral mills because of crust formations, consequent clogging and blow-back issues, which amplify the inconsistency of the output PSD.
   - Inconsistent inlet Particle size distribution may lead to inconsistent final result, and the need for batches to be mixed at the end the campaign to obtain an homogeneous particle size.
   - Certain pigments or formulations may be highly abrasive and excessively consume certain parts of the spiral jet mill, such as the venturi tube or the micronization chamber.

Schedio has developed a series of solutions to address and prevent crust formation inside the milling chamber.

2. The issues that arise when using mechanical mills (roller mills, pin mills, etc.) for cosmetic powders:
   - Cleanliness of the system;
   - Heating of the product, which cause changes in certain products' pigmentation;
   - These types of mills have numerous moving mechanical components, which are subject to wear and require frequent check-ups in order to assure an acceptable uniformity between different production batches;
   - Frequent maintenance, abrasion and wear of mechanical and hydraulic parts, can cause frequent and relatively long-lasting downtimes;
   - Safety risks: mechanical mills pose safety risks to operators, if the equipment is not adequately maintained or errors are committed during the process;
   - Noise and vibrations.

PROCESS IMPROVEMENT WITH THE OPPOSITE JET MILL…HOW DOES IT WORK TO IMPROVE THE MICRONIZATION PROCESS?

The Opposite Jet Mill, unlike the Spiral Jet Mill, does not generate a Vortex inside the milling chamber, but rather concentrates the energy of the compressed air in a central point at the base of the mill.

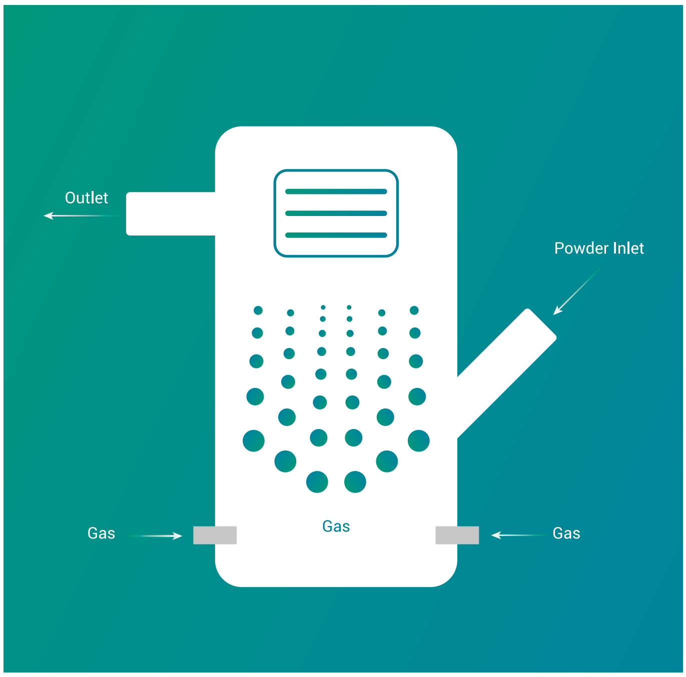
An opposite jet mill, also known as a fluidized bed jet mill, is a type of mechanical mill used for micronization of various materials, including cosmetic powders. The working mechanism of an opposite jet mill is based on a principle of fluidization, where a fluid (usually air) is used to create a fluidized bed of the material to be milled. The fluidized bed is created by introducing compressed air into the milling chamber through a set of nozzles or jets. The compressed air creates a high-speed stream of air that entrains the particles of the material to be milled, lifting them up and suspending them in the fluidized bed.

The fluidized bed is then subjected to high-speed collision between the particles, which causes the particles to break down into smaller particles. The size of the particles can be adjusted by adjusting the pressure and flow rate of the compressed air, as well as by adjusting the distance between the nozzles.

The milled particles are then separated from the fluidized bed by a classifying wheel, which separates particles based on their size. The larger particles are returned to the fluidized bed for further milling, while the smaller particles are collected for further processing.

The advantages of the Opposite Jet Mill in cosmetic applications include:

- Narrow particle size distribution: Opposite jet mills can produce a very narrow particle size distribution, which is especially useful for cosmetics and other applications where particle size uniformity is critical.
- Low-heat process: Jet milling is a low-heat process, which helps to preserve the integrity of heat-sensitive materials, such as pigments and vitamins, during the milling process.
- Adaptability: Opposite jet mills can handle a wide range of materials and particle sizes, making it adaptable to different cosmetic formulations.
- Scalability: Opposite jet mills can be scaled up or down depending on production needs, which can help to increase or decrease production capacity as needed.
- Safety: Opposite jet mills are relatively safe to operate, as they do not generate ultrafine particles or have many moving parts that can pose a risk to operators.
- Consistent particle size distribution: Due to the classification system, the outlet particle size distribution will be the same, regardless of the initial particle size distribution.
- Easy feeding: Opposite jet mills do not require precise feeding, as they have load cells that keep the amount of product inside the chamber constant.
- Dust-free operation: Opposite jet mills are considered a relatively clean process when properly connected to a cyclone filter, preventing dust generation.
- The opposite jet mill has been used on multiple trials on cosmetic products, including the pure red7 test described in this white paper, each of them with optimal particle size distributions.
- Further trials have to be carried out in order to properly verify the operability of the opposite jet mill on cosmetic products, as well as to develop specific solutions for the most common challenges described above which, in certain cases, may be applicable to the opposite jet mill as well.
- We do already know however that it is possible to easily grind extremely abrasive products, for example minerals such as granite or more specifically for cosmetics, mica and talc in their coarsest forms to obtain a micronized product that can satisfy different cosmetic applications. What remains to be further studied and improved, is the behaviour with particularly oily or fatty ingredients or cosmetic formulations, which RED7 is a good example of.



TRIAL RESULTS

The RED7 pigment is a particularly oily product, currently not possible to be micronized with a spiral jet mill (even when optimized for oily substances), which is widely used as raw ingredient for lipsticks. The current approach developed by Omnicos is to mix the raw ingredient with a binder, and mill it through a roller mill, multiple times, to obtain the desired particle size distribution.

The purpose of this test was to verify the micronization efficacy of the opposite jet mill on the pure RED7, without binding agents.

We had reason to believe that the fluidized bed generated by the process gas in the opposite jet mill could help overcome issues caused by the stickiness of the RED7 pigment. The purpose of the trial was to obtain a omogeneus, micronized particle size distribution, without clogging challenges.

A total of three runs has been carried out, the first two, to verify the final particle size distribution, and the third one (long run), to verify the productivity.

The process parameters that have been used during the first two trials were:

Trial 1:
1) Grinding pressure: 5 bar
2) Classifier rotation speed: 8'000rpm

Trial 2:
1) Grinding pressure: 7 bar
2) Classifier rotation speed 12'000 rpm

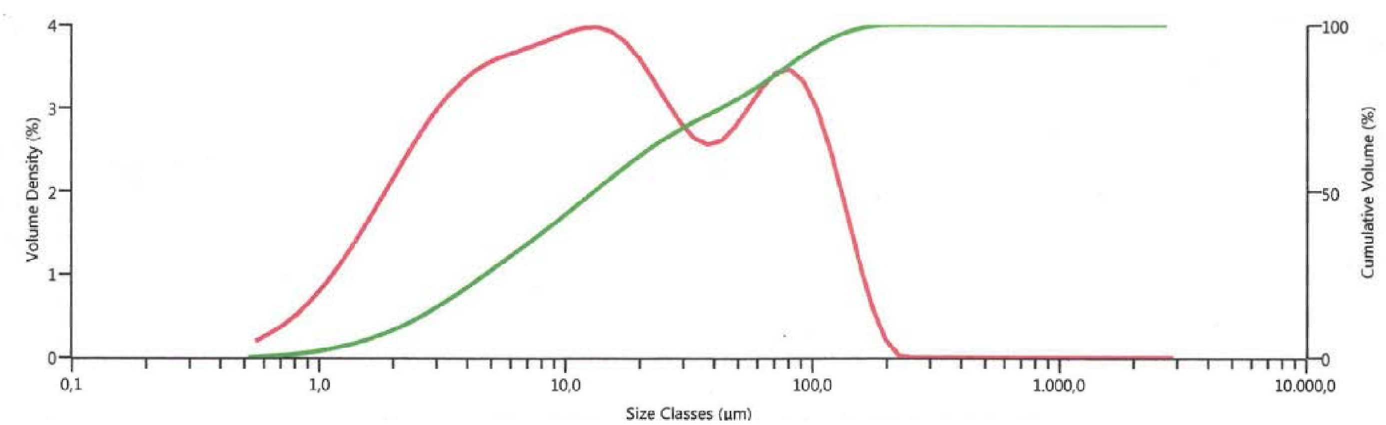
Other parameters, set and adjusted during the trails were: net product range and air barrier pressure.

As the jet mill is on load cells, once the net product inside the milling chamber goes below the set net product range, the jet mills commands the feeding sequence to introduce raw material. This translates in the productivity not being a pre-set parameter, but rather being a consequence of the other parameters set above. .e.g in case of higher classifier speed, the residence time of the product inside the milling chamber raises, and therefore the productivity reduces as consequence.

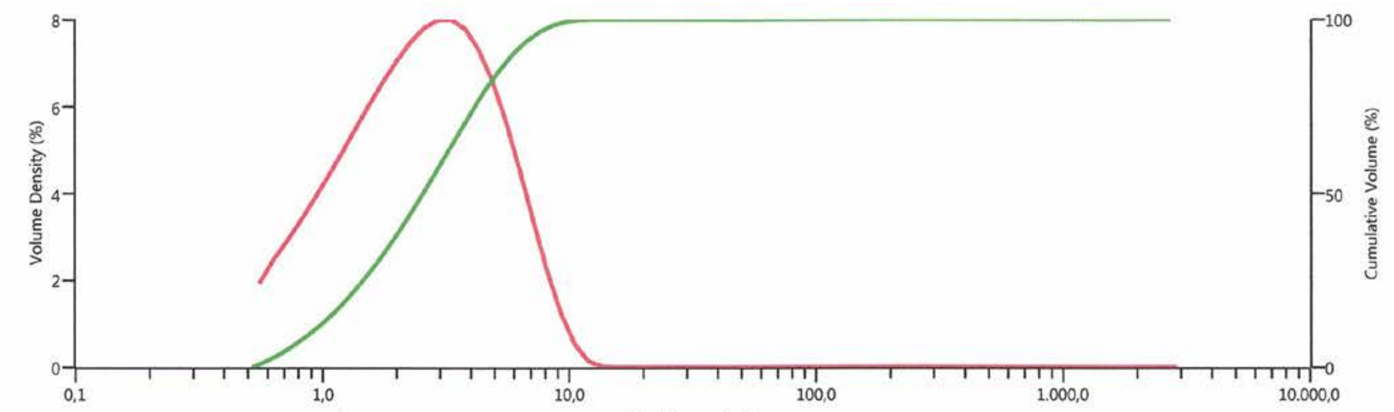
The product has been successfully micronized, with an homogeneous particle size distribution (see table1), that satisfied the application necessities.

	D10	D50	D90	D100
Raw material	2.25 µm	13.1 µm	88.6 µm	211 µm
Trial 1 result	0.9 µm	2.56 µm	5.95 µm	12.7 µm
Trial 2 result	0.95 µm	2.71 µm	5.85 µm	12.7 µm

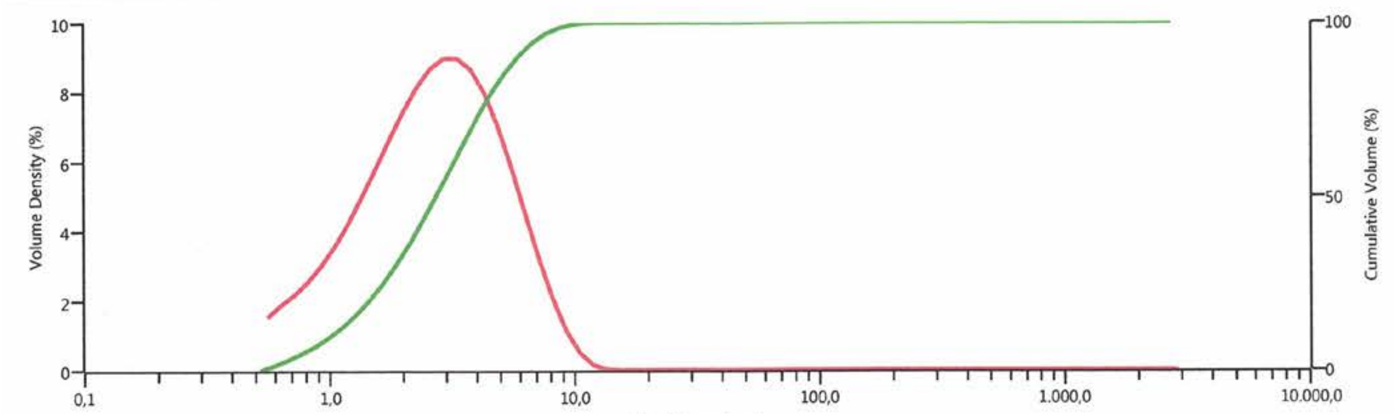
RAW



TRIAL 1



TRIAL 2

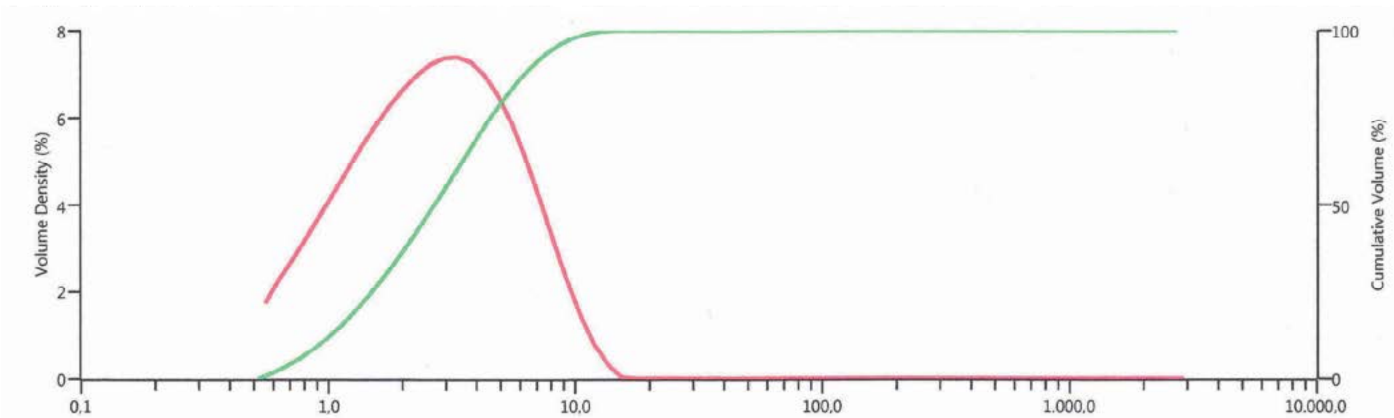


During the third trial (long run), the following parameters have been used:

Trial 3:
1) Grinding pressure: 7 bar
2) Classifier rotation speed 8'000 rpm

	D10	D50	D90	D100
Raw material	2.25 μm	13.1 μm	88.6 μm	211 μm
Trial 3 result	0.9 μm	2.69 μm	6.76 μm	14.4 μm

TRIAL 3



By reducing the classifier speed, and keeping a 7 bar grinding pressure, it has been possible to obtain still a fine and homogeneous particle size distribution, but with a higher throughput of 350 gr during a 1 hour of continuous micronization.

Oily and sticky products in general have the tendency to clog the micronization chamber over time due to residuals layering. The amount of residuals remaining inside the milling chamber and layering over time varies by product to product. This poses a particular threat within cosmetic industry in particular, due to the nature of the processed products. It was therefore key to determine the success of the trials, to verify also the behaviour of the RED7 inside the milling chamber and the formation of layers over time, which could lead to machine downtime.

At the end of the trials, on the RED7, little to no residues have been found stuck inside the milling chamber.

The amount of residues, remained constant throughout the trials, suggesting that the product tends to create a thin layer on the stainless steel surfaces, which does not interfere with the micronization and classification process and does not increase over time.

Such residues, have been estimated to be 190grams total, which are generated in the first 20 minutes, and remained constant for the rest of trial 3 (long run).

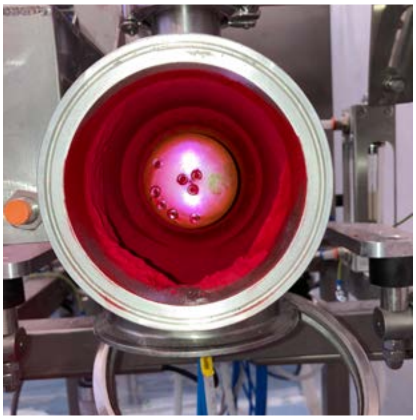


Fig. 1: Top of the mill

Fig. 2: Bottom of the jet mill

Fig. 3: Classifier and outlet

CONCLUSIONS

In conclusion, the use of the Opposite Jet Mill has proven to be an effective solution for the micronization of pure RED7 pigment, commonly used in the cosmetics industry. By utilizing this technology, the clogging problems typically found when using spiral jet mills and mechanical mills have been overcome, resulting in an homogeneous particle size distribution without the need for binding agents. Furthermore, the Opposite Jet Mill offers a low maintenance, all-in-one solution that combines both milling and dynamic classification of powder, with a lack mechanical moving parts requiring frequent maintenance, making it a consistent machine for a wide variety of powders at different grades of micronization. However, further tests should be carried out to improve efficiency and verify that the amounts of residues inside the micronization system remain constant over longer runs.

This will provide valuable insights into the long-term performance of the opposite jet mill and its ability to consistently produce high-quality, micronized RED7.

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