

Solving challenging caking behavior and poor flowability of nutraceutical blends with porous minerals

Fabian Doering and Carolina Diaz Quijano



Case Study

The power of powders in nutraceuticals

Powders play a key role in the nutraceutical industry, offering unique benefits. They are versatile and can be incorporated into various galenic forms – such as sachets, granules, tablets, capsules, and beverages – making them an ideal choice in product development, ensuring both flexibility for manufacturers and ease of use for consumers. Additionally, powdered botanical ingredients preserve the full vegetal matrix, retaining all natural constituents and improving the bioavailability and efficacy of active ingredients through the synergistic potential of phytoactives. Processing techniques like spray drying further enhance the stability, solubility, and shelf life of powdered ingredients, ensuring their long-term efficacy. Compared to liquid formulations, powders have a lower risk of microbial contamination and a longer shelf life. Their concentrated nature and lower weight can also make them more cost-effective to produce and transport, resulting in potential cost savings for both manufacturers and consumers.

Overcoming flow and caking challenges

However, powders present challenges in both industrial and home conditions. Due to particle interactions, they may flow poorly and cake over time, leading to manufacturing disruptions. For example, powders that flow poorly or cake can cause uneven mixing, leading to content uniformity issues. They may also clog dosing systems, resulting in costly downtimes for cleaning. Additionally, they may cause inconsistent die filling resulting in unacceptable variations in tablet and capsule weight, as well as defects such as tablet capping or lamination, or both. Further, working with challenging powders can cause excessive wear on equipment due to sticking and clogging, while also creating complications in packaging and downstream powder management.

The challenges associated with powders stem from the interactions between their particles. Factors such as surface roughness which increases friction, irregular particle shapes that lead to mechanical interlocking, electrostatic charges that cause particles to clump, or surface moisture that results in adhesion through capillary bonding all contribute to poor flow and caking.

Incorporating glidants and anticaking agents

Because each powder behaves as a distinct and intricate matrix, these issues are difficult to manage. While process interventions such as vibrating equipment or temperature and humidity control can address some of these challenges, they are often costly, inefficient, and insufficient. The most widely used solution for improving flow and preventing caking is the incorporation of glidants and anticaking agents. These ingredients reduce friction and mechanical interlocking by smoothing the surface of the base powder particles, decrease cohesion by coating them, and prevent adhesion by absorbing moisture. (Figure 1)

Typically, glidants and anticaking agents include precipitated silica, calcium and potassium silicates, talc, tricalcium phosphate, calcium carbonate, magnesium carbonate, and magnesium oxide. Some manufacturers also use stearic acid or magnesium stearate, known for their lubricant properties, to enhance powder flow.

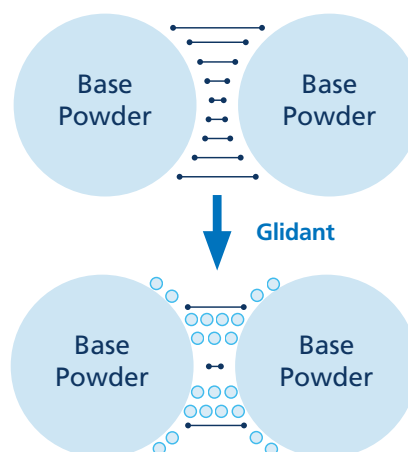


Figure 1. The interactions between powder particles (represented by violet links) are reduced by adding glidants and anticaking agents (shown as yellow circles).

Innovating with porous calcium carbonate

Omya has long been present in powder processing with its Omya Calcipur 110, a natural calcium carbonate designed to reduce friction, mechanical interlocking, and cohesion – improving powder flow. Thanks to its tailored particle size distribution, Omya Calcipur 110 acts as a separating agent. However, due to its mineral composition and micron-sized particles, it cannot absorb moisture and therefore prevent adhesion and caking.

The challenge was therefore to enable moisture absorption without reducing the particles to a nano-scale size. The solution was to engineer the calcium carbonate to make it porous, allowing it to trap moisture and oily components within its micron-sized particles through capillary forces. To validate its effectiveness, Omyanutra 300, our porous calcium carbonate, underwent three tests across various powder formulations. These demonstrated its effectiveness in improving powder flow and preventing caking.

Product	Calcium carbonate (%)	Tribasic calcium phosphate (%)	d50 (µm)	Surface area (m ² /g)	Loose bulk density (g/ml)	Oil absorption (g/100g)
Omyanutra 300-OG	53	47	6.6	53	0.13	150
Omyapharm 500-OG						

Figure 2. Omyanutra 300 and Omyapharm 500 are highly porous minerals composed of calcium carbonate and tribasic calcium phosphate. Their fine pore network, making up approximately 80% of the particle, allows for excellent moisture binding and high oil absorption. The only difference between the two is their documentation package—Omyapharm 500 includes a DMF (Drug Master File), while Omyanutra 300 does not.

Test 1: Echinacea extract

We tested an echinacea extract, known for its immune-boosting properties but also for its poor flowability and compactability during tableting.

- **Flowability:** Adding 1% Omyanutra 300 significantly improved flow, achieving results similar to precipitated silica (Figure 3). However, increasing the concentration to 2% or 4% did not further improve the angle of repose.

- **Compactability:** Despite improved flow, the echinacea extract alone could not be compressed into tablets. To assess its impact on compactability, we blended the extract with 50% microcrystalline cellulose (MCC). Unlike silica, adding 1% Omyanutra 300 significantly improved tablet hardness at low compression forces (Figure 4).

- **Tableting Process Stability:** Omyanutra 300 also stabilized the tableting process (Figure 5), reducing variability in tablet hardness (by 30%), weight (by 46%), and compression force (by 12%).

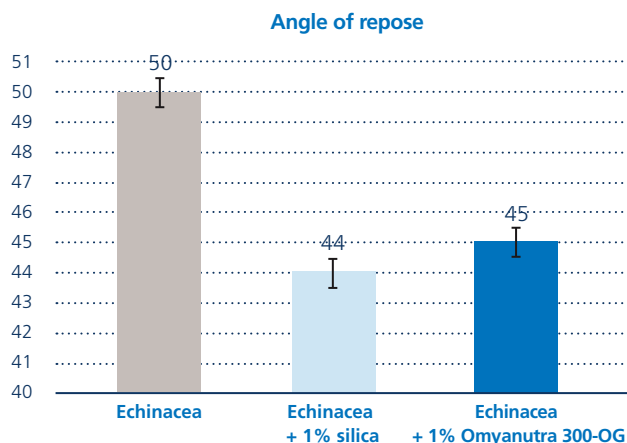


Figure 3. Angle of repose of an Echinacea extract and MCC blend (50:50) with and without flow and anticaking agents. The addition of Omyanutra 300 significantly reduces the angle of repose of the Echinacea extract.

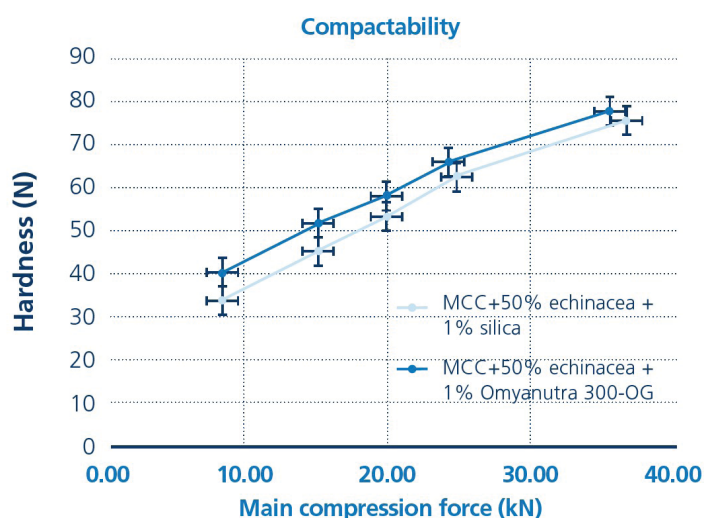


Figure 4. Compactability profile of an echinacea extract and MCC blend (50:50) with and without flow and anticaking agents. Omyanutra 300 significantly increases tablet hardness at low compression forces compared to silicon dioxide.

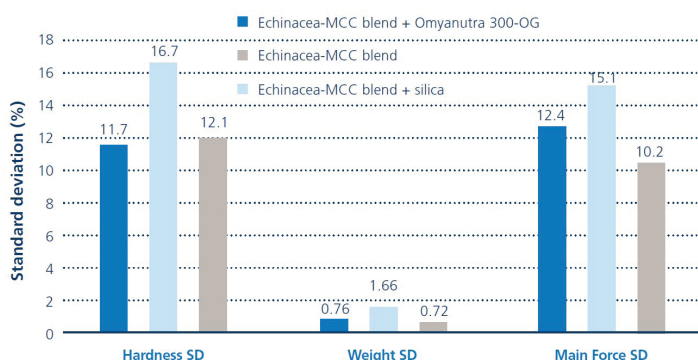


Figure 5. Standard deviations of key tableting parameters of an echinacea extract and MCC blend (50:50). The addition of 1% Omyanutra 300 reduces standard deviations in tablet hardness, weight, and compression force by 30%, 46% and 12%, respectively.

Test 2: Turmeric powder

We tested turmeric powder, a well-known anti-inflammatory ingredient, to assess the impact of Omyanutra 300 on its properties.

- **Flowability:** Adding 1% Omyanutra 300 or silicon dioxide significantly reduced the angle of repose (Figure 6). Increasing the concentration to 2% provided only a slight additional benefit, reducing the angle to 33% (vs. 35%).

- **Caking Behavior:** We evaluated caking after 6 and 9 days at 25°C and 75% relative humidity (Figure 7). The addition of anticaking agents helped reduce caking compared to the control.

- **Compactability & Tableting Stability:** While compactability tests on the turmeric-MCC blend (50:50) showed no major improvement over silicon dioxide (Figure 8), Omyanutra 300 significantly improved tableting process stability. It reduced standard deviations in tablet hardness (by 11%), weight (by 35%), and compression force (by 20%) (Figure 9).

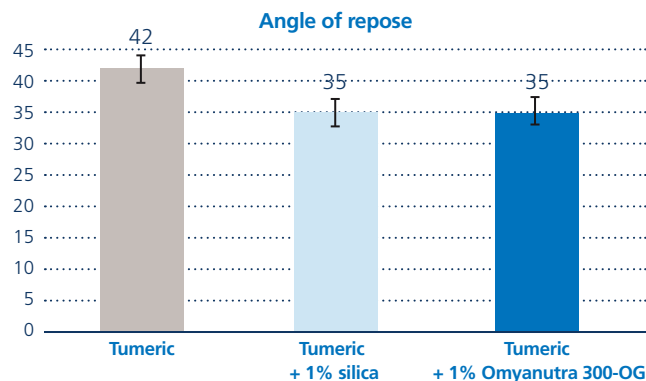


Figure 6. Angle of repose of a turmeric powder with and without flow and anticaking agents. Omyanutra 300 significantly reduces the angle of repose of the turmeric powder.

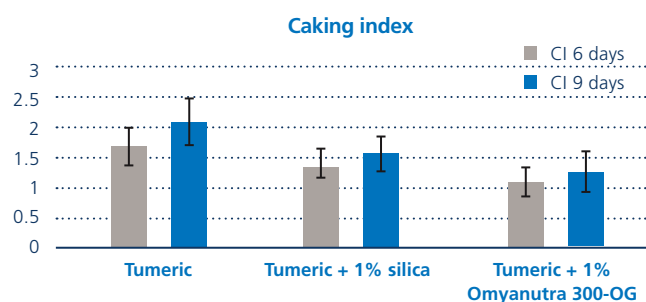


Figure 7. Caking behavior of turmeric powder after 6 and 9 days at 25°C and 75% relative humidity. Unlike silica, Omyanutra 300 significantly reduces caking compared to turmeric powder alone, particularly after 9 days.

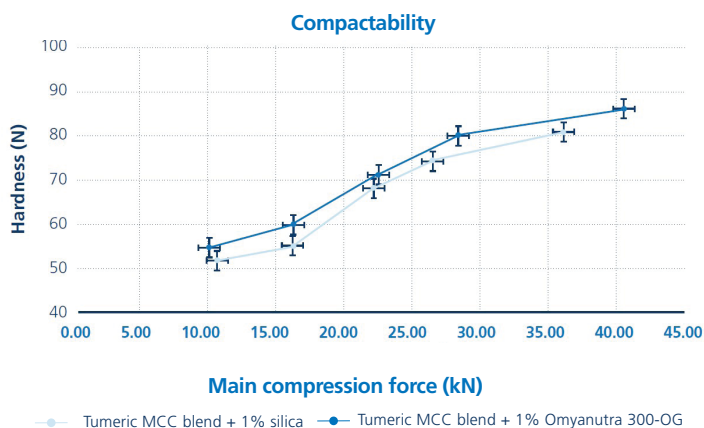


Figure 8. Compactability profile of a turmeric powder and MCC blend (50:50) with and without flow and anticaking agents. Omyanutra 300 increases tablet hardness at low compression forces compared to silicon dioxide, but the difference is not significant.

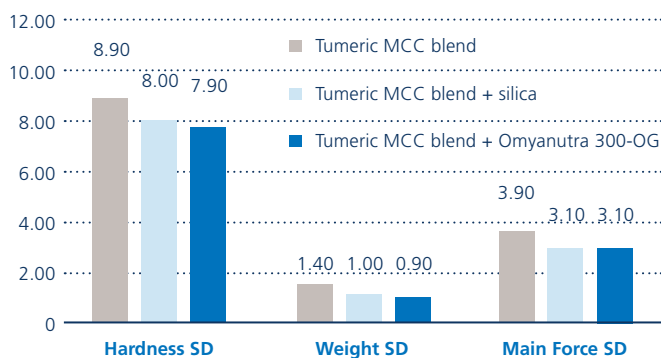


Figure 9. Standard deviations of key tableting parameters for a turmeric powder and MCC blend (50:50). The addition of 1% Omyanutra 300 reduces standard deviations in tablet hardness, weight, and compression force by 11%, 35% and 20%, respectively.

Test 3: Creatine-containing pre-workout blend

We tested a pre-workout blend containing creatine and examined the impact of adding Omyanutra 300.

- **Flowability:** Adding 1% Omyanutra 300 significantly reduced the angle of repose (Figure 10). However, increasing the concentration to 2% or 4% did not lead to further improvement.

- **Caking Behavior:** The blend was stored at 25°C and 75% relative humidity for 2 hours. Without additives, it fully caked, forming a 33mm crust. Adding 1% Omyanutra 300 reduced caking by nearly 50% compared to the blend alone and by 30% compared to the blend with 1% silica (Figure 11).

- **Compactability & Tableting Stability:** While Omyanutra 300 did not enhance compactability in a pre-workout blend + MCC (50:50) compared to silica, it significantly improved process stability. It reduced standard deviations in tablet hardness (by 11%), weight (by 31%), and compression force (by 14%) (Figure 12).

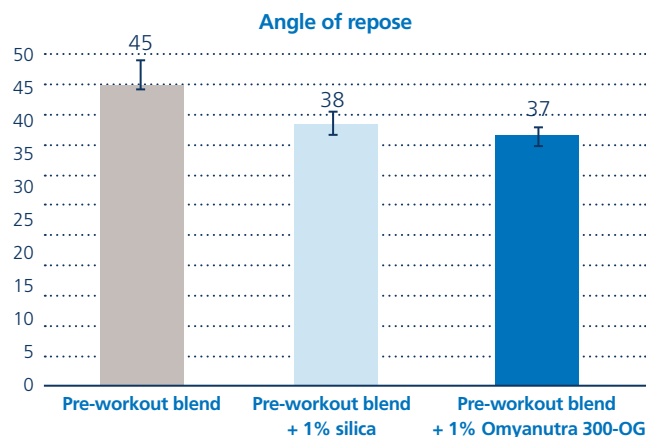


Figure 10. Angle of repose of a creatine-containing pre-workout blend with and without flow and anticaking agents. Omyanutra 300 significantly reduces the blend's angle of repose.

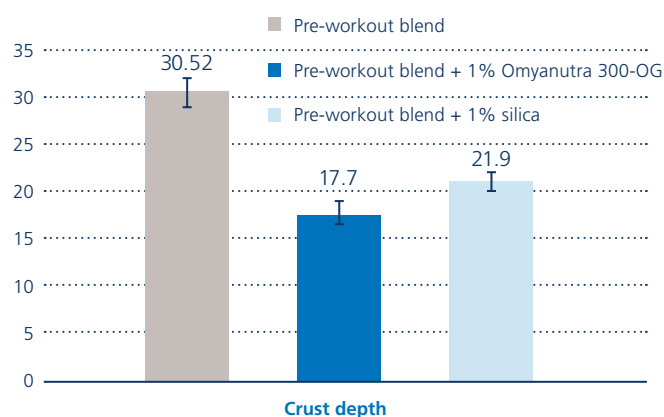


Figure 11. Caking behavior of a creatine-containing pre-workout blend after 2 hours at 25°C and 75% relative humidity. The addition of 1% Omyanutra 300 reduced caking by nearly 50%, while silica reduced it by 30%.

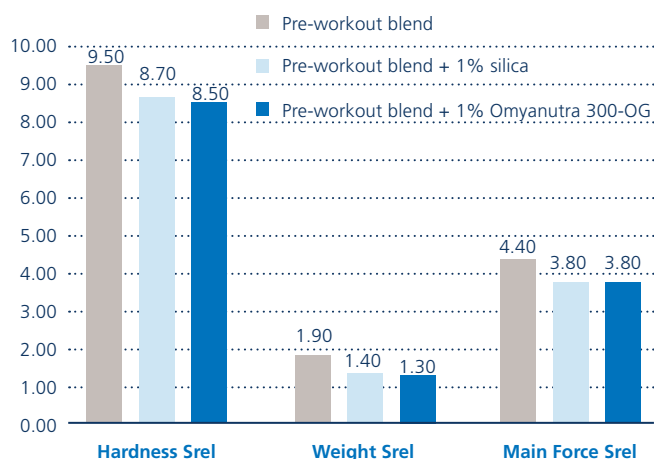


Figure 12. Standard deviations of key tableting parameters for a creatine-containing pre-workout blend and MCC blend (50:50). The addition of 1% Omyanutra 300 reduced standard deviations in tablet hardness, weight, and compression force by 11%, 31%, and 14%, respectively.

Conclusion

All three tests demonstrated that Omyanutra 300 can significantly improve the flow of poorly flowing nutraceutical blends and reduce caking in most cases. Improving the flow offers significant advantages for downstream processes, facilitating transport, mixing, dosing, filling, packaging, tableting, and encapsulation. Minimizing caking extends the storage time of blends before use and/or maintains flow under varying temperature and humidity conditions. In terms of compactability, while Omyanutra 300 at a 1% addition only significantly improves tablet hardness in certain blends, it generally helps stabilize the tableting process. It achieves this by significantly reducing variability in key parameters such as tablet hardness, weight, and compression forces. A more stable tableting process improves tableting speed, increasing the output of a press, and, thus, reducing costs. Further, it helps minimize equipment wear, contributing to longer machine lifespan and lower maintenance costs.

Beyond its performance, Omyanutra 300 is a non-nano, fully digestible material that contributes to dietary calcium intake. This makes it a safer alternative to nano particles, which may accumulate in the body and pose potential health risks. Additionally, Omyanutra 300 generates less dust than other alternatives, enhancing manufacturing safety and efficiency by minimizing cleaning time. Omyanutra 300 is a food-grade material composed of GRAS-certified substances and is manufactured in France under FSC22000 standards. For pharmaceutical applications, a pharma-grade version, Omyapharm 500, is also available.

In conclusion, Omyanutra 300 stands out as a superior alternative to conventional flow enhancers and anticaking agents, improving the nutritional profile of powders and reducing risk, thanks to its non-nano size, digestible nature and low dusting properties.

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