MICROTRAC
PARTICLE CHARACTERIZATION

Synergistic approaches in Zeta Potential, Particle size and Stability Analysis for advanced research
Case study on a cosmetic formulation

Context

Formulators encounter various challenges daily, such as the impact of switching suppliers on product quality, the risk of formula destabilization when substituting a component, or the need to incorporate a new ingredient. To meet customers' and formulators' expectations for the final product, it is crucial to employ cutting-edge technologies that comprehensively characterize systems. MICROTRAC provides a wide range of technologies and tools for characterization, supporting formulators in the product development process for fast decision-making based on facts. This note presents a case study that combines zeta potential, particle size, and shelf-life stability to optimize a sunscreen formulation.

Three instruments, ONE conclusion

MICROTRAC provides technologies with unique features:

STABINO ZETA

For automated fast zeta potential measurements at high concentration.

NANOTRAC FLEX

For particle size distribution **without dilution.**

TURBISCAN

For **1000x faster** final formulation destabilization detection and quantification without dilution

By combining all these technologies, the time needed to introduce products to the market is reduced and the optimal formula is identified quickly, with the highest level of accuracy.

How it works?

- **STABINO ZETA**

STABINO ZETA offers a **reliable** zeta potential measurement based on streaming potential even on high concentration system. Its smart **mix-and-measure** operation prevents sedimentation, ensuring **accurate results**. Additionally, the **built-in titration function** enables fast determination of the isoelectric point (IEP) for samples with particle size from **0.3 nm to 300 µm** and concentration from **0.01 to 40 vol%.**

- **NANOTRAC FLEX**

NANOTRAC FLEX is the innovative Dynamic Light Scattering (DLS) in a **unique probe design** for particle size distribution **without dilution**. It allows **fast and reliable** measurements directly in any vessel without needing a specific sample cell. For an accurate particle size distribution measurement between **0.3 nm to 10 µm,** up to **40 w%** of particles**.**

- **TURBISCAN**

TURBISCAN is the world reference stability analyzer based on Static Multiple Light Scattering (SMLS). This advanced technology enables fast detection of any kind of destabilization **without dilution 1000x faster** than the naked eye. It accurately **detects and quantifies** sedimentation, creaming, and flocculation in product ranges of **0.0001 to 95 v/v%.**

More about Microtrac's instruments

Case Study

Understanding these three parameters simplifies decision-making to overcome formulation issues.

For this note, sunscreen formulation is studied as an example. A lower zeta potential correlates with larger particle sizes and increased formula instability impacts UV protection.

By combining these three parameters, the most effective sunscreen is formulated with optimal stability and UV protection. The study follows the following three steps:

1. **Raw Material Selection** based on **zeta potential** and **particle size analysis**
2. **Enhance UV efficiency** after selecting the best filter
3. **Stability analysis** of the new sunscreen formulation

1. Raw material selection

Before formulating, the most suitable UV filter must be selected. We're looking for particles with high zeta potential with the smallest particle size.

- **Zeta potential**

To guarantee UV protection, three particles are studied in water: TiO_2, ZnO, and $CePO_4$. The zeta potential measure (Table 1) shows the closest value to the neutral value for $CePO_4$. This one is more likely to agglomerate and reduce UV protection.

Particle	Zeta Potential (mV)
TiO_2	-50,5
ZnO	-18,8
$CePO_4$	-8,8

Table 1: Zeta potential measures

- **Particle size distribution**

Figure 1 shows the particle size distribution measurements. For the $CePO_4$ we directly see the impact of its low zeta potential, resulting in the formation of two distinct particle populations, which indicates the tendency to agglomerate. In contrast, despite higher zeta potential, ZnO has also a large particle size which can lead to a lower efficacy against UV-radiation.

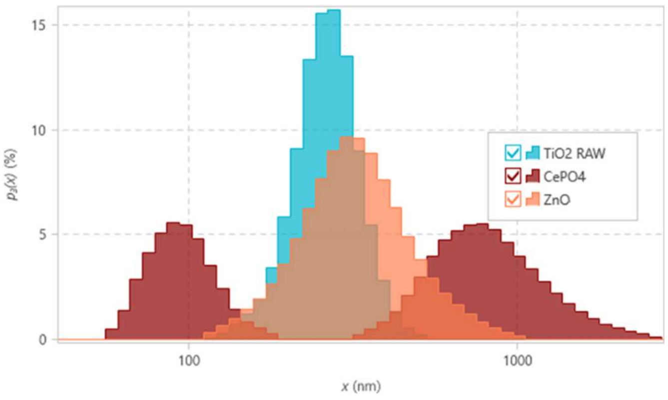

Figure 1: Particle size distribution

The TiO_2 particles, have an ideal zeta potential and particle size, making them the most suitable raw material for this formulation.

2. Enhance UV-efficiency

Some inorganic particles, such as the TiO_2 we have selected, have high photocatalytic activity, which can degrade the formulation. To overcome this, the particles can be chemically modified to increase colloidal stability and reduce photocatalytic activity.

- **Particle Characterization**

To reduce photocatalytic activity, the surface of the TiO_2 previously selected is modified with silica particles. Zeta potential measurement is a fast method to confirm this particle modification. An acid titration is performed to determine their isoelectric point, IEP (Figure 2).

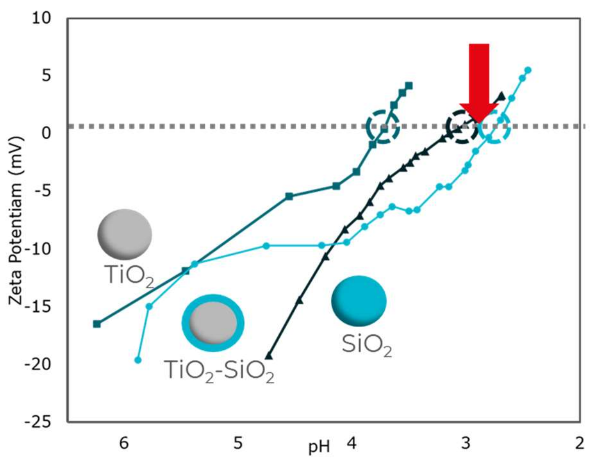

Figure 2: Isoelectric point of particles to evaluate surface modification

As we see in the graph, the IEP of the coated particles is close to the silica's IEP, which means that the TiO_2 particles were well covered by silica. The IEP of coated particles has decreased, these particles are more resistant to pH variations.

Since we modified the particle's surface following the IEP measurement, it's necessary to reevaluate particle size. Figure 3 shows that the coated particles are larger than the raw TiO_2.

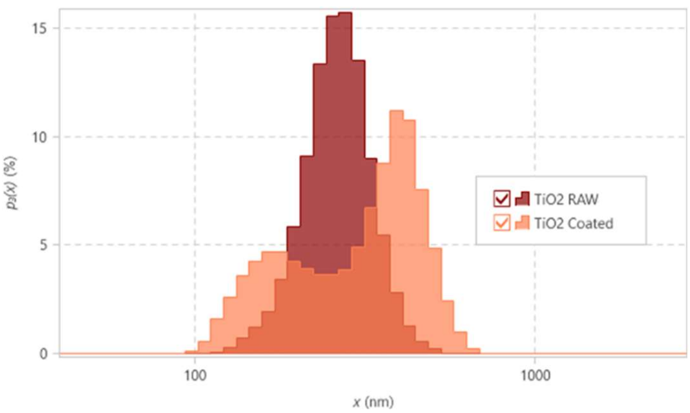

Figure 3: Coated particle size distribution

Although the zeta potential remains relatively close, it's essential to determine if this size difference will impact the formula's stability.

3. Destabilization detection

Now that the pre-formulation steps are done, the final sunscreens are formulated to investigate the stability with TURBISCAN.

The destabilization detected by TURBISCAN are represented as a function of sample height (in mm) over time. The bottom of the sample is represented on the left part of the graphic and the top of the sample is on the right part. The color gradient on the time scale corresponds to each scan time lapse with the first scan in blue and the last scan in red. The recorded backscattering level is put in "Delta Mode"- ΔBS as we see in Figure 4.

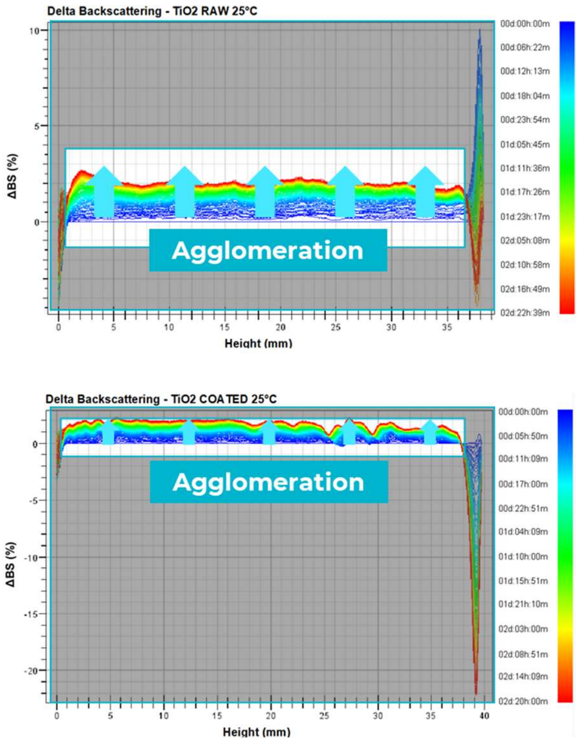

Figure 4: Destabilization detected by TURBISCAN

We observe an increase in BS signal over time in the middle of the sample for both sunscreens indicating an agglomeration. However, this agglomeration is less important in the formula with the coated particles which means less impact on the final sunscreen's SPF.

The agglomeration is identified within the first hours of measurement before the destabilizations become visible to the naked eye. Figure 5 shows the samples at the end of the analysis.

Figure 5: Destabilization not detected by naked eye

- **Sunscreens stability and TSI**

On top of identification, to rank the best formulation, the TURBISCAN Stability Index (TSI) is used. The TSI is an automatic calculation that sums all destabilizations into a single number for easy, one-click ranking and comparison. The higher the TSI value, the lower the Stability.

Figure 6 shows the TSI kinetics with the sunscreens formulated with raw and coated TiO_2. "A" represents the sunscreen formulated with raw TiO_2 at 25°C and 54°C, and "B" the sunscreens formulated with the coated TiO_2 at the same temperature range.

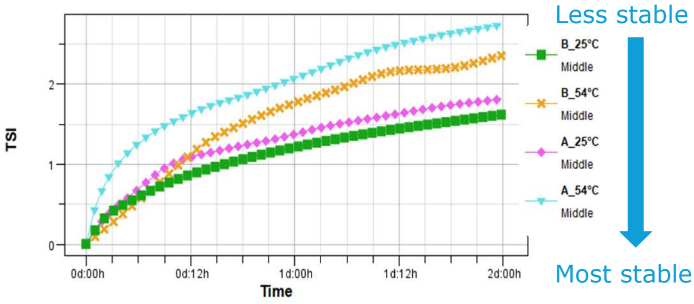

Figure 6: TSI kinetics over 2 days

The sunscreen with the coated TiO_2 is the most stable formulation, identified within the first hours of measurement, and is also less impacted by temperature variations.

Summary: Three Instruments, One conclusion

STABINO ZETA, with its online titration capability, allowed us to accurately characterize the raw materials and the modified particles.

NANOTRAC FLEX provided accurate results of particle distribution to identify the particle with the best size for the formula.

TURBISCAN has detected destabilization mechanisms faster than the naked eye to identify the most stable formulation.

Zeta Potential, Particle Size and Stability measurements guided us towards the optimal formulation. All the measurements were done **without dilution** and gave **fast results** with **reliable** technologies.

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