BET-Surface Area as key parameter for discrimination between Nano- or Conventional material

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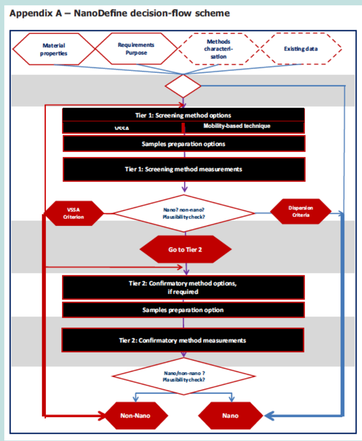
Introduction

EU definition for classification of nanomaterials usually requires extensive investigation via high-resolution electron microscopy followed by elaborate data treatment to ensure that a product is or is not a nanomaterial. Following the discussion about potential risks of those materials, producers in various application fields are urged to check their product portfolio for potential nanomaterials and take further actions for risk assessment – or to change product properties accordingly. For food ingredients, European Food Safety Authority (efsa) has published in July 2018 a "Guidance on risk assessment of the application of nanoscience and nanotechnologies in the food and feed chain", which includes a decision scheme for general discrimination between nano- or conventional material. This decision scheme is based on "volume-specific surface area method" (VSSA) as a screening method and relies on the knowledge of the skeletal density, the geometrical nature about the material itself - and the specific surface area via BET-method.

EU-Nanodefine: Decision Flow Scheme

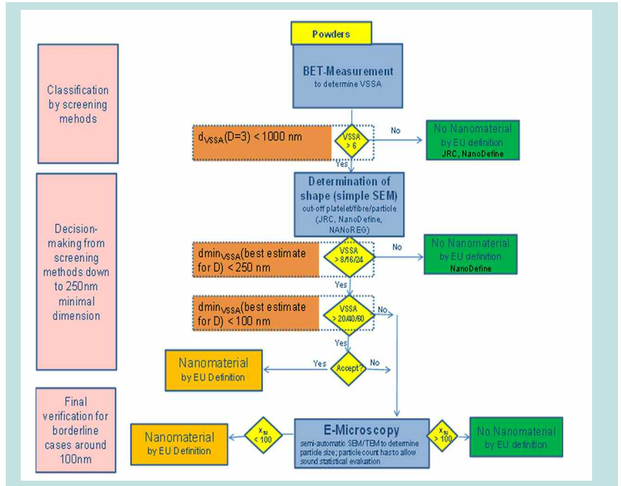
https://www.efsa.europa.eu/en/efsajournal/pub/5327 page 79



BET-Surface-area / VSSA – a direct decision criterion!

EU-Nanodefine: VSSA - Specific Flow Scheme

https://link.springer.com/content/pdf/10.1007%2Fs11051-017-3741-x.pdf page 61



Volume-Specific Surface Area (VSSA):

VSSA = BET-Surface Area x Skeletal Density [m^2/ml]

- Any sample: not „nano" if VSSA < 6 m^2/ml

Otherwise - depending on shape:

Morphology	not „nano" if VSSA is...	nanomaterial if VSSA is...
Platelet:	< 8 m^2/ml	> 20 m^2/ml
Fibre:	< 16 m^2/ml	> 40 m^2/ml
Particles:	< 24 m^2/ml	> 60 m^2/ml

If value is in between cut-off-values: further investigations are neccessary...

In case of porous media: outer surface area has to be evaluated...

Example: Organic Acid-Salt (platelet)

Start: VSSA = 16.3 m^2/ml => no clear decision...

BET-Surface Area = 8.32 m^2/g

Skeletal Density = 1.96 g/ml => VSSA = 16.3 m^2/ml

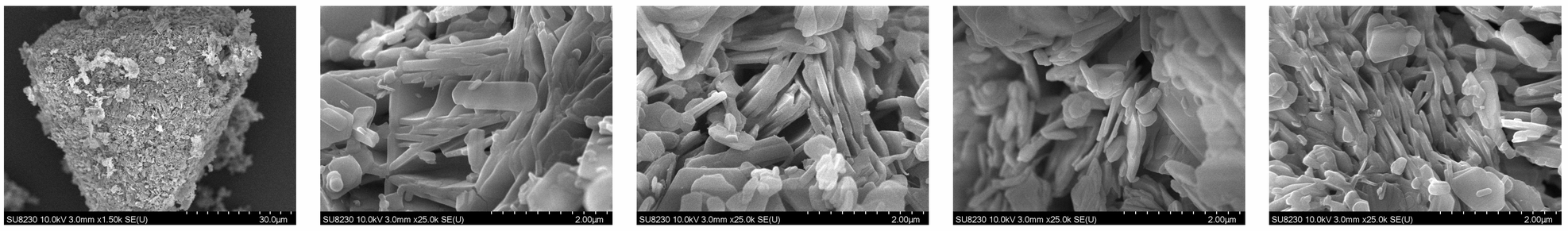
HR-SEM: 85 % in between 1-100 nm (Ø 69 nm plate-thickness) => nanomaterial.

=> variation of production parameters:

VSSA = 7.2 m^2/ml => conventional material!

BET-Surface Area = 3,67 m^2/g

Skeletal Density = 1.96 g/ml => VSSA = 7.2 m^2/ml



HR-SEM: 33 % in between 1-100 nm (Ø 128 nm plate-thickness) => conventional material.

Conclusion

Although visualizing methods are inevitable in first stage and also skeleton density has to be investigated (for example via He-Pyknometry), following the EU-decision scheme by relying on BET-Surface area measurements fastens up the decision process and helps especially for product development – like to drag a product out or into the nanomaterial-range – relatively fast determination of specific surface area determination can be used to check for helpful variations of process parameters.

Nanomaterial-Definitions...

ISO (2015): material with any external dimension on the nanoscale ('nano-object') or having an internal or surface structure in the nanoscale ('nanostructured material')

EU (2011): A natural, incidental or manufactured material containing particles, in an unbound state or as an aggregate or as an agglomerate and where, for 50 % or more of the particles in the number size distribution, one or more external dimensions is in the size range 1 nm - 100 nm.

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Literature

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Wendel Wohlleben & Johannes Mielke & Alvise Bianchin & Antoine Ghanem & Harald Freiberger & Hubert Rauscher & Marion Gemeinert & Vasile-Dan Hodoroaba, Reliable nanomaterial classification of powders using the volume-specific surface area method; **J Nanopart Res (2017) 19: 61**