

Effect of Drying Methods on the Particle Morphology of Microcrystalline Cellulose

Annina Lähdeniemi * and Olli Dahl

Microcrystalline cellulose (MCC) is a purified partially depolymerized nonfibrous form of cellulose, a crystalline powder composed of porous particles. In this study, the drying of MCC was investigated with two different solids content MCCs by using three different drying methods: a high-velocity cyclone dryer, a spray dryer, and a fluidized bed dryer. The effects of the different drying techniques on the geometrical dimensions and morphology of the dried MCC particles and aggregates were researched. Based on the results, the dried MCC particle morphology is highly dependent on the used raw material properties as well as the liquid removal mechanism during drying. The preserving of the morphology of the raw material MCC was mostly achieved by fluidized bed drying, which facilitated MCC to dry as discrete particles with high surface area and lesser aggregation when using the MCC with 45% dry consistency as raw material. Spray drying was observed to produce small, most circular particles with the most homogenous size distributions and high velocity cyclone largest, most heterogenous and irregular shape particles and aggregates. All results have been presented as such after drying without screening.

DOI: 10.15376/biores.20.4.9524-9541

Keywords: Microcrystalline cellulose; MCC; Cellulose drying; Cyclone dryer; Spray dryer; Fluidized bed dryer; Cellulose powder; Never-dried MCC

Contact information: Department of Bioproducts and Biosystems, Aalto University School of Chemical Technology, PO Box 16300, Vuorimiehentie 1, Espoo, Finland;

* Corresponding author: annina.lahdeniemi@aalto.fi

INTRODUCTION

Microcrystalline cellulose (MCC) is a purified partially depolymerized nonfibrous form of cellulose derived mostly from wooden plants and cotton with mineral acids. It is an amorphous, odorless, tasteless, thermally stable crystalline powder composed of porous particles. MCC is capable of forming stable colloidal suspensions, creating a three-dimensional network, and maintains these properties as well at high temperature as in low pH. Consequently, MCC is highly applicable in products that require solution stabilizers, bulking agents, material texture modifications, compaction stability, dry processability, compatibility, and other material modification properties (Battista and Smith 1962; Vasiliu-Oprea and Nicoleanu 1993; Krawczyk *et al.* 2009; Tuason *et al.* 2012).

The first products from MCC were developed and patented for the food and pharmacy industries during the 1960's. At present, these industry sectors are yet today MCC's most common application areas with cosmetics. MCC has been widely used in industrial applications as a suspension stabilizer and reinforcing agent for final products. Since MCC is inert for humans, it can be widely applied in pharmaceuticals, where it is used currently as bulk, binder, compression aid additive for tablets and thickening, and as

a structural agent for healing creams. Even today, it is considered as most possibly the best dry filler-binder currently available and remains the major excipient for the preparation of pharmaceutical pellets by extrusion/spheronization (Balaxi *et al.* 2009). Additionally, MCC presents a high dilution potential, making it suitable for the formulation of low dose and potent drugs. Possessing about 35% market share, it has been estimated that the pharmaceutical sector is the biggest application area for MCC (Transparency market research 2015).

The physical properties of MCC differ plenty based on the used application. The average particle size of commercial MCC products ranges from 20 to 250 μm , although larger agglomerate products between 1000 and 1400 μm are available as well (FMC 2015; JRS 2015; MingTai 2015). Different processing conditions affect the properties of MCC in terms of surface area, porosity, crystallinity, molecular weight, moisture content, and shape. These differences also affect the powder and compaction behavior of the MCC. By changing particle size and aspect ratio, porosity, surface charge, adding functional groups, or by changing the drying circumstances and creating bundles of several single MCC particles, the properties of the processed tablets can be adjusted (Mills 2007; Kamel *et al.* 2008; Thoorens *et al.* 2014).

Because MCC drying requires high energy input, it is desirable to have a simple and economical dryer. Commercially, spray drying is one of the most commonly used techniques in the industry for drying MCC products into powder form. In the past, different fluidized bed dryers, spin flash dryer as well as lyophilization have been among the available options for MCC drying (Beck *et al.* 2012; Peng *et al.* 2012). One promising future drying technology that has been investigated, but not yet been used on MCC, is the high velocity cyclone or vortex type dryer (Nebra *et al.* 2000). The different drying methods have dissimilar and distinct effects on the particle morphology, size, porosity and aggregation, thus affecting the characteristic of the produced dried product and its post-processing procedures, such as screening and milling.

The main advantages of spray drying are the extremely short contact time of the suspension with the heat source and elimination of post-processing stages for fiber separation, such as grinding and screening. Additionally, spray drying increases the sterility of the product and different high-quality powders can be produced by engineering the size, shape, and morphology of particles by optimizing the operating conditions. The disadvantages of the process are clogging the spraying systems with above-scale material and the possible formation of particle clusters. Most MCC dried by the spray drying imparts a high inter-particulate porous and aggregate nature to the dried powder (Walton and Mumford 1999; Cal and Sollohub 2010; Peng *et al.* 2012; Amin *et al.* 2014; Paudel *et al.* 2013).

Fluidized bed drying (FBD) facilitates powders to dry as discrete particles with a low inter particulate void (Nwachukwu and Ofoefule 2020). The advantages of fluidized bed drying include high rate of moisture removal, high thermal efficiency, easy material transport inside the dryer, straightforward control, and low operating and maintenance costs (Mujumdar and Devahasthin 2003). Challenges of this technology are the entrainment of fine particles, attrition or pulverization of particles, along with agglomeration of fine particles as well as achieving a suitable gas-solid contact, bed stability and uniform product quality for certain types of FBDs (Daud 2008; Liu *et al.* 2014; Zimmermann *et al.* 2016).

The results of high velocity cyclone drying depend on the equipment design, but as a non-thermal separation technology, cyclones have the advantages of a high separation

efficiency, low energy consumption due to the relatively low temperatures used, and simple maintenance (Grimm *et al.* 2017; Dehdarinejad and Bayareh 2023).

Typically, a highly desirable feature of a good drying technique is the preserving of the properties and morphology of the MCC particles. It is well-established knowledge that drying has a permanent effect on the cellulose structure. Upon the drying of cellulose, the water removal leads to the formation of internal hydrogen bonds due to the presence of hydroxyl groups on the cellulose surface, which promotes an attraction among the cellulose fibers. This phenomenon, which promotes the formation of fiber agglomerates and the loss of their original dimensions in the nanometric scale, is commonly known as hornification (Spence *et al.* 2010). Hornification is known by the literature as an irreversible or partially reversible bond between the hydroxyl groups present in the cellulose and it has been reported that hornification decreases MCC's binding ability during tableting as well as swelling and disintegration (Balaxi *et al.* 2009, Diniz *et al.* 2004). New studies made by Sjöstrand *et al.* (2023) shows that the hornification process begins already at a level of dryness of 20 percent and at temperatures as low as 40 °C. Nevertheless, the biggest changes in fiber structure are seen at temperatures above 100 °C.

Considering MCCs multiple current applications and future potential ones, it is of interest to investigate the possibility of producing MCC with different shape in cross-section to increase the surface area as it is of great importance for specific applications. To the authors' knowledge, there has been no research data published of drying MCC with high velocity cyclone. Additionally, no research have been made of the drying of never-dried MCC produced with AaltoCell™ technique that varies from the commercially available MCC manufacturing methods with its low chemical consumption and lower production costs (Vanhatalo 2017). It is expected that the dried MCC powder quality and particle morphology are highly dependent on the used raw material as well as the liquid removal mechanism and the liquid-MCC interaction. Therefore, a study of different drying methods including their qualitative evaluation and analysis of the produced powders is thought to give an insight into the processing of the materials involved.

In this study, unmodified, never-dried microcrystalline cellulose MCC (produced with patented AaltoCell™ method, PURA™ MCC, Nordic Bioproducts Group) as a raw material was dried with three different drying methods; fluidized bed, high velocity cyclone and spray drying. The main aim was to discover how the various drying methods affect physical properties of the dried MCC powder. Due to the absence of additives and nano-size particles, the manufactured powders should be highly applicable for food additives and drug delivery. The effects of the drying actions on the crystalline structure, morphology, and geometrical dimensions of the manufactured dried MCC powders were investigated.

EXPERIMENTAL

Cellulose Raw Materials and the Production of MCC (AaltoCell™)

The cellulose raw material RA45% was never-dried microcrystalline cellulose manufactured with the AaltoCell™ method, as described in Vanhatalo and Dahl (2014). In brief, bleached softwood kraft pulp was hydrolyzed with a 1.5% dosage of sulfuric acid at 160 °C at 10% consistency for 110 min using a tube-like reactor with volume of 2.5 dm³. After the hydrolysis, the reactor was cooled, and the MCC material was washed with distilled water in a Büchner funnel until the washed filtrate conductivity was below 5.0

$\mu\text{S}/\text{cm}$. The washed MCC material was centrifuged at 4500 rpm with a filter bag to the dry consistency of 45%. The other MCC raw material, RA60%, was pre-dried from RA45% MCC with a heated Lödige Ploughshare® Laboratory Mixer (Gebrüder Lödige Maschinenbau GmbH, Germany) to the dry consistency of 60%.

Experimental Equipment

High-velocity cyclone dryer

The two MCC samples (45% and 60%) were processed with a cyclone dryer as described in Grimm *et al.* (2017). A schematic flow sheet of the process is presented in Fig. 1. An electric motor-powered fan creates a high-velocity airstream guided into an air channel and mixed with the processed material. The processed raw material is fed into the inlet airstream with a tubular disc conveyor rod. From the air channel, the mixed material flows into a cyclone-formed device in which the changes in radial velocity and pressure produce heavy vibrations in the cellulose material, causing them to dehydrate and disintegrate. Fluids and the finest fraction of the processed material (reject) escape from the cyclone through the upper part of the cone and are sent into a separate baghouse filter unit. The dried product comes out from the bottom of the cyclone.

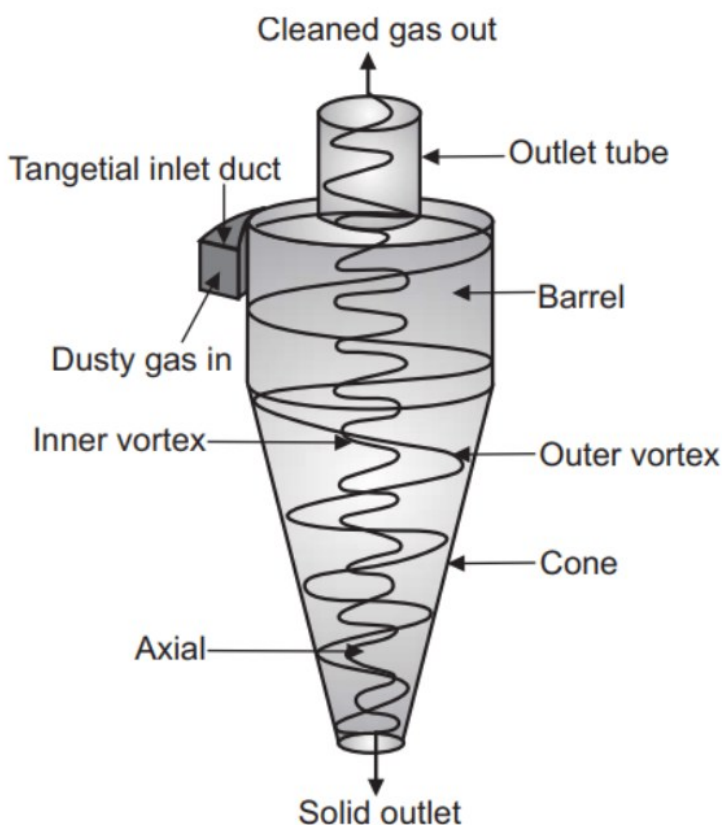


Fig. 1. Simplified illustration of the high velocity cyclone drying process. Obtained from the Industrial Mechanical separators and vessel's internet site. (Source: Industrial Separation GmbH and Co, 2025)

In test trials, the intake air was heated from 12 to 60 °C with a heat exchanger. During the experiment, the fan and the conveyors were set to run at approximately the same time that the intake air was preheated. The feeding rate was 4 kg/h, and the temperature

inside the cyclone was 49 °C. None of the parameters or functions of the drying process (such as input air temperature, material input rate, depth of the upper shutter, *etc.*) were optimized or maximized during the experiment.

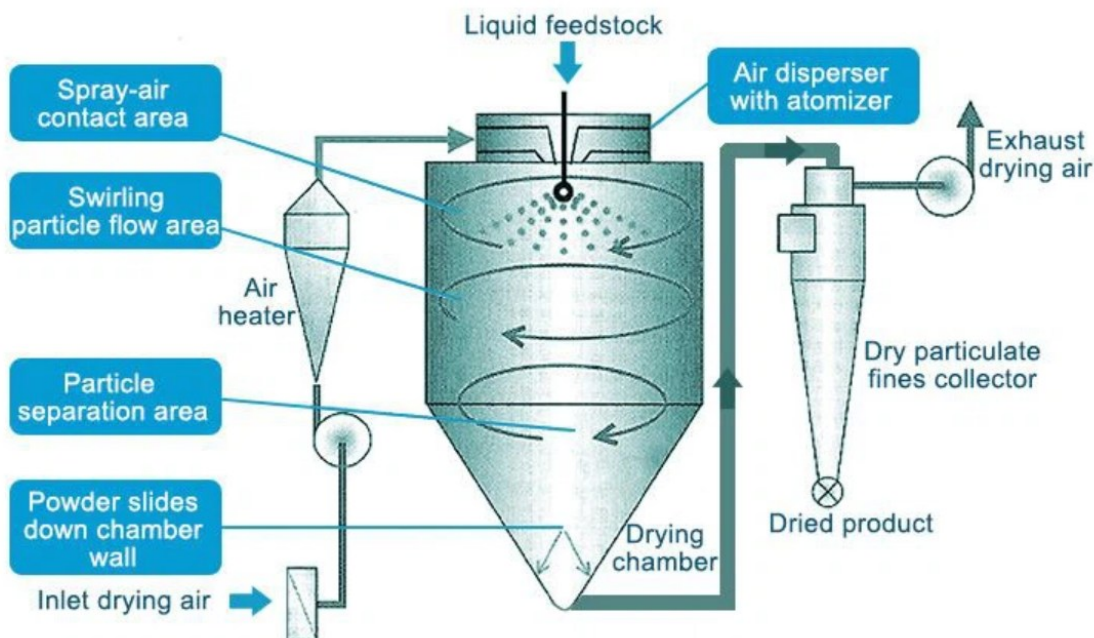


Fig. 2. Simplified scheme of the Spray drying process obtained from the manufacturer's internet site. (Source: Toption Decive Ltd and Co, 2025)

Spray dryer

The produced MCC was converted to dry powder by spray drying (Niro Mobile Minor, Niro Atomizer Ltd., Copenhagen, Denmark), using inlet and outlet air temperatures of 350 and 90 °C, respectively. Both MCC samples (45% and 60%) were diluted into 5% solid feed consistency before the drying. The spray drying process, presented in Fig. 2, can be characterized by three central stages: (a) in the first phase, the fluid, consisting of the water suspension and cellulose, is dispersed by spraying in the form of droplets; (b) in the second phase, the suspension droplets becomes in contact with the warm air stream as the heat transfer begins; and (c) in the third step, the solvent evaporation and formation of solid particles (Oliveira and Petrovick 2009).

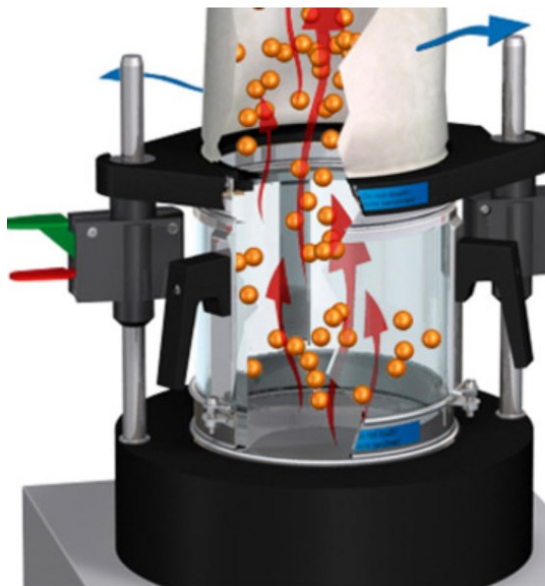


Fig. 3. Illustrated picture of the particle fluidization in the Retsch fluidized bed laboratory dryer obtained from the manufacturer's internet site. (Source: Retsch GmbH and Co, 2024)

Fluidized bed drying

A fluidized-bed dryer (rapid dryer model TG 100, Retsch GmbH and Co., Germany), with bed column of cylindrical shape approximately 18 cm in diameter and 22 cm high, was used for the drying experiments. The two raw materials, MCC 45% and MCC 60%, were dried at a temperature of 60 ± 1 °C and inlet air of 30 m³/min for 3 h. The fluidized drying process in the used laboratory device is displayed in Fig. 3. In fluidized bed drying, hot air is blown through the perforated plate, where the hot air gets distributed. The air pressure causes the fluidized particles to float. Heat and mass are exchanged effectively between particles and the hot air due to the large surface area of the particles. Violent mixing of the particles takes place in the fluidized layer because of gas bubbling (Hovmand 2020). The hot air stream extracts moisture from the particles and then exits through the filter bag in the cover of the equipment.

Test Standards and Methods

The particle size distribution, particle circularity, as well as the aggregate sizes and shapes of the samples were measured with Mastersizer 2000 equipped with a Hydro 2000MU dispersion and insight units (Malvern Instrument Ltd, Worcestershire, UK) that uses ultrasonication to disperse and separate the particles. The size distribution results and circularity plots as well as the particle agglomerates' shape images were obtained with the Mastersizer 2000 software version 5.12. The measurement was conducted as published (Vanhatalo and Dahl 2014). Particle size distribution d(0.1), d(0.5), and d(0.9) values were reported to describe physical particle sizes. All particle size distribution and circularity measurements were done in triplicate.

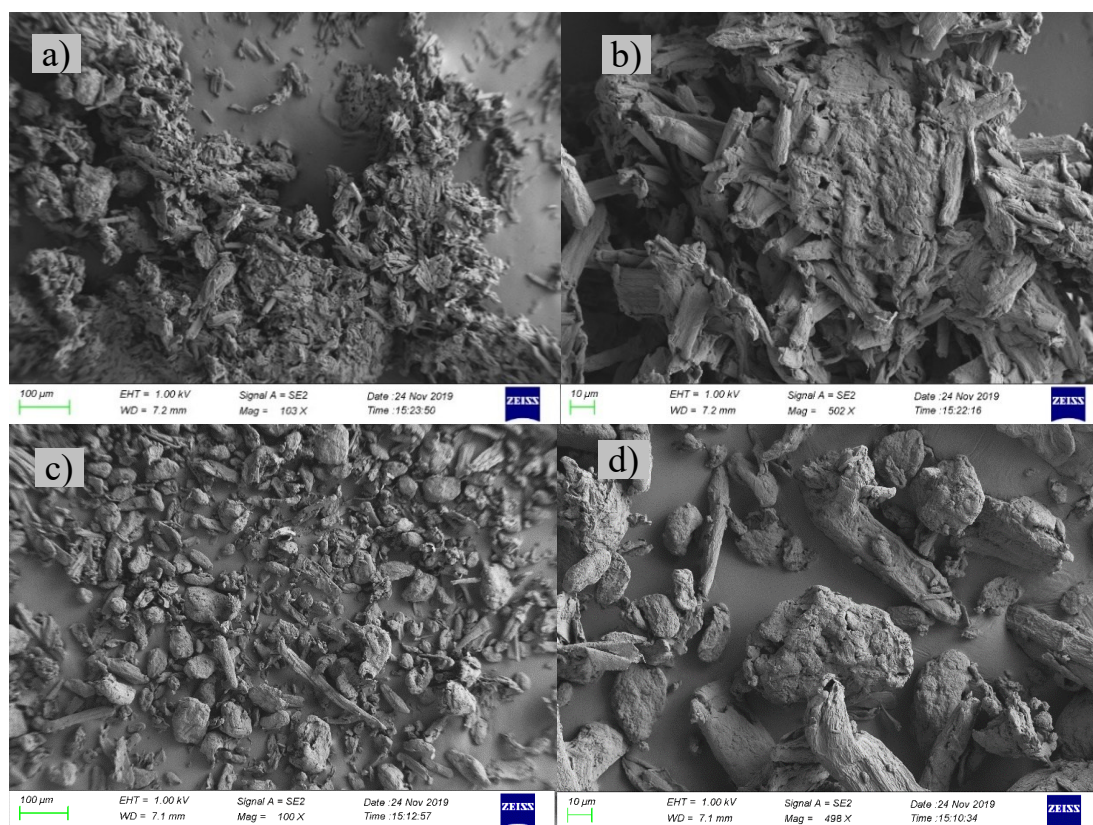
The structural morphology of the CelRaw and hydrogel samples were observed with scanning electron microscopy (Zeiss Sigma-VP, Jena, Germany) at 1.5 keV, equipped with an Everhart-Thornley detector. Prior to imaging, the samples were sputter coated (Emitech K-100X, Lewes, UK) with a 10-nm gold layer to obtain proper conductivity.

RESULTS AND DISCUSSION

Particle Morphology with SEM Micrographs

SEM investigation was used to inspect the particle surface morphology. The SEM micrographs of the dried samples and raw materials are shown in Figs. 4 and 5. The micrographs show that the two raw materials displayed dissimilar shapes and apparent size differences. The never-dried raw material with the dry consistency of 45% presented clear crystalline cellulose structure with sizable, aggregated particles with more specific surface area. The pretreated raw material with the dry consistency of 60% appeared to have already less crystalline, more separated, spherical particles with reduced accumulation and smooth surfaces.

With the never-dried raw material, it seems that the crystalline structure of the particles and the specific surface area is maintained only with fluidized-bed drying, whereas spray drying and high velocity cyclone produced larger circular particles with less available surface area. In addition, it appears that the formation of aggregates was greater with the high velocity cyclone and spray drying than with the fluidized bed. These observations from the SEM micrographs are consistent with those reported by other researchers in past.



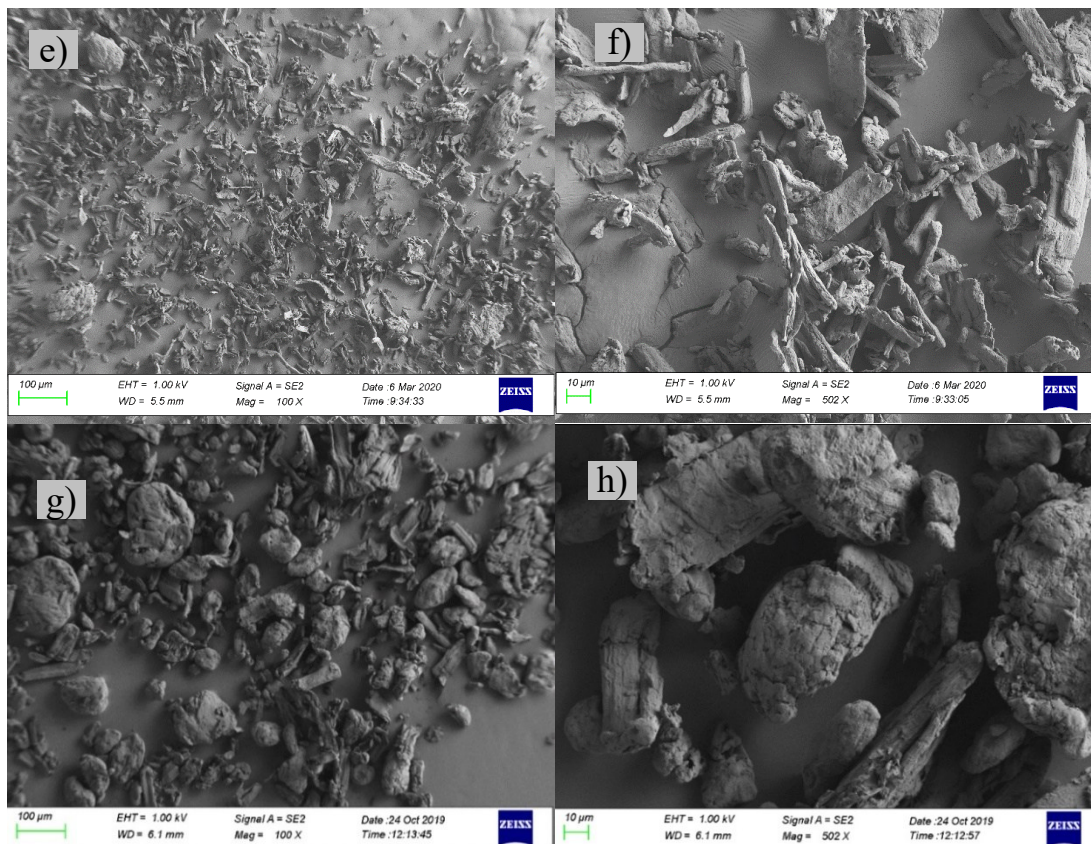


Fig. 4. SEM micrographs with two different magnifications (100x and 500x) of the never-dried MCC raw material a) and b), and the samples dried from it with spray dryer (Spray 45%) c) and d), fluisized bed dryer (FDB 45%) e) and f), and high-velocity cyclone dryer (HVC 45%) g) and h)

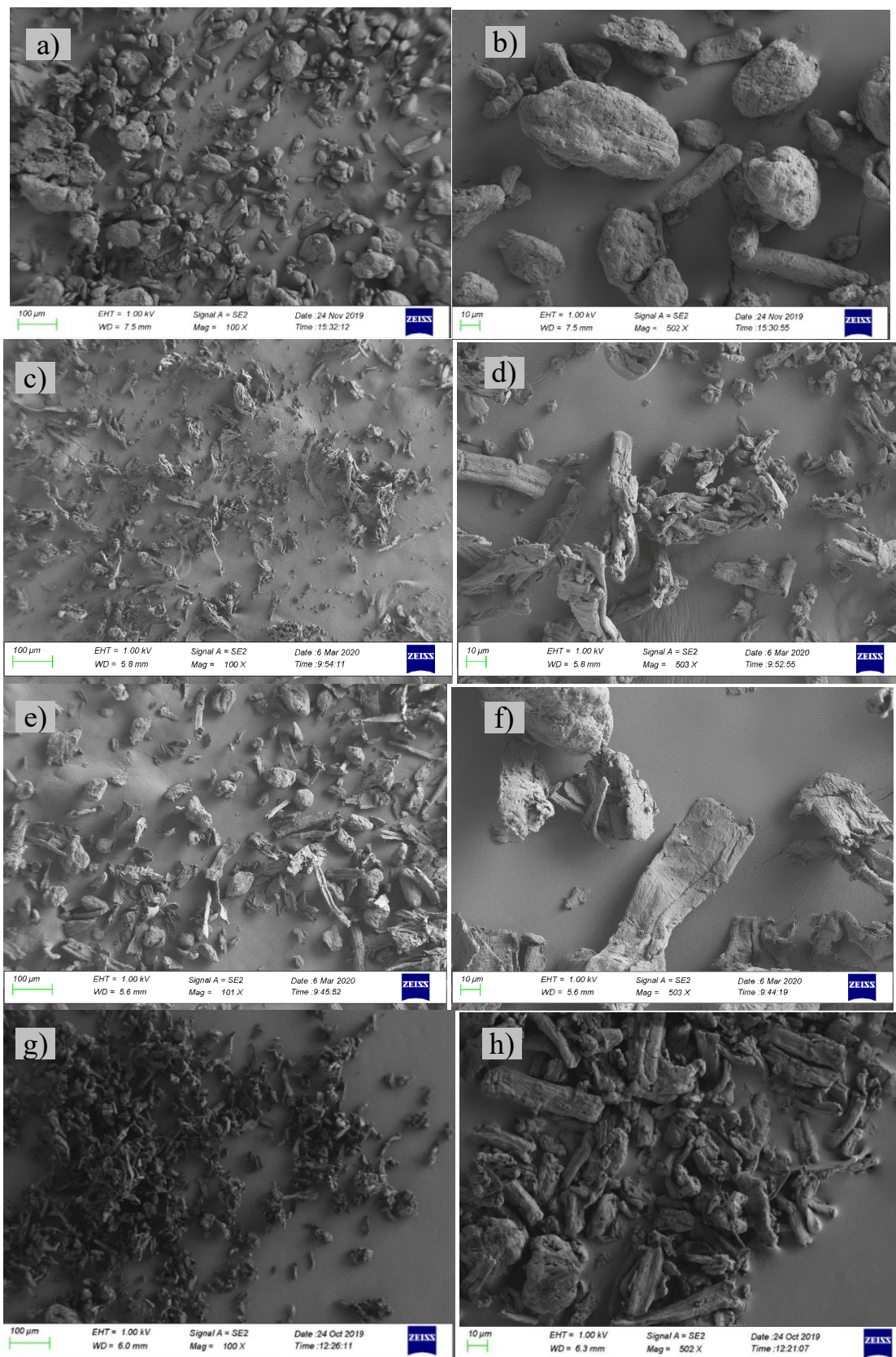


Fig. 5. SEM micrographs with two different magnifications (100x and 500x) of the pretreated MCC raw material a) and b), and the samples dried with spray dryer (60%) c) and d), fluisized bed dryer (FDB 60%) e) and f), and high-velocity cyclone dryer (HVC 60%) g) and h).

Nwachukwu and Ofoefule (2017) described that MCC powder dried with fluid bed was found to have single particles and lesser aggregation. Zimmermann *et al.* (2016) stated that the fluid bed drying method aids powders to dry as discrete particles with a low inter particulate void. In various studies, spray drying has been found to form bead-like aggregates consisting of rod-shaped particles (Pandey *et al.* 2012; Haafiz *et al.* 2014; Zimmermann *et al.* 2016; Ioelovich 2019).

With the pre-treated raw material, the different drying methods showed very different particle morphologies compared to the never-dried raw material. As shown in the SEM images shown in Fig. 5, it seems that the spray drying and high velocity cyclone produced tinier and less aggregated particles than the fluidized-bed. This drying behavior is completely opposite compared to the one with the never-dried raw material. Here however, the spray dried sample demonstrated more the high inter-particulate porous nature described previously by Haafiz *et al.* (2014) and Pandey *et al.* (2012) as well as the irregular, elongated, plate-shaped particles, and irregular aggregate structures reported by Amin *et al.* (2014) compared to the one dried from the never-dried MCC raw material. It seems that the pre-dried, already fairly hornified raw material has stronger particle clusters and aggregates that needs a more powerful separation technique inside the drying equipment. This may be the reason why the gentler drying method, fluidized bed appears to have the largest aggregates when using the pre-dried MCC as raw material. The SEM micrographs imply that the drying methods are highly affected by the dry consistency of the raw material and the different drying techniques produce diverse particle morphologies and surface textures. This is possibly due to the different drying behavior of the drier, partially hornified fiber particle structure that is stiffer with more closed surface structure.

Particle Size Distributions

The particle size distributions $d(0.1)$, $d(0.5)$, and $d(0.9)$ are parameters that indicate the percentage of particles 10%, 50%, and 90% possessing diameters equal to or less than the given value. The distribution values of the two raw materials were 41 μm , 96 μm and 184 μm for the never-dried MCC (RA45%) and 11 μm , 26 μm and 60 μm for the pretreated MCC (RA60%), respectively. The RA60% particles were reduced already in size due to hornification taken place during the water removal in the pretreatment. It appears that even the 15 % increase of the dry consistency of the raw material reduced the particle size *circa* 70% in all the particle distributions.

The results of particle size measurement of the dried samples are presented in Fig. 6. When using the never-dried raw material (RA45%), the particle size was reduced *ca.* 85% in all distributions with spray and fluidized-bed drying and *ca.* 75% with high velocity cyclone. With the pretreated raw material (RA60%) drying, the situation was quite opposite. Here, the high velocity cyclone achieved the greatest decrease, 32 to 52% reduction in particle size distributions. The spray drying reached 25 to 43% decline in the particle sizes with different distributions with RA60%, while the fluidized-bed drying led to only 26% decrease in the particle size in the two lesser size distributions, $d(0,5)$ and $d(0,1)$, while the sizes in distribution $d(0,9)$ increased with one percent. Most surprisingly, the particles dried with fluidized-bed were considerably larger in the bigger distributions $d(0,9)$ and $d(0,5)$ with the RA60% compared to RA45%. With the other drying methods, the particle size in $d(0,9)$ and $d(0,5)$ were 40 to 50% smaller with RA60% compared to the ones produced with RA40%.

When comparing the different drying techniques, the greatest reduction of particle sizes in all distributions was achieved by the fluidized-bed with the never-dried MCC,

RA45%, and by spray drying with the pretreated MCC, RA60%. The largest particle sizes were produced in all distributions by high velocity cyclone with the never-dried raw material and by the fluidized-bed with the pretreated raw material. The spray drying lowered all particle sizes most equally implying that it produced most homogenous dried product. When considering the size distribution results, it appears that the high velocity cyclone produced the most heterogenous particle sizes, which was also seen in the SEM micrographs.

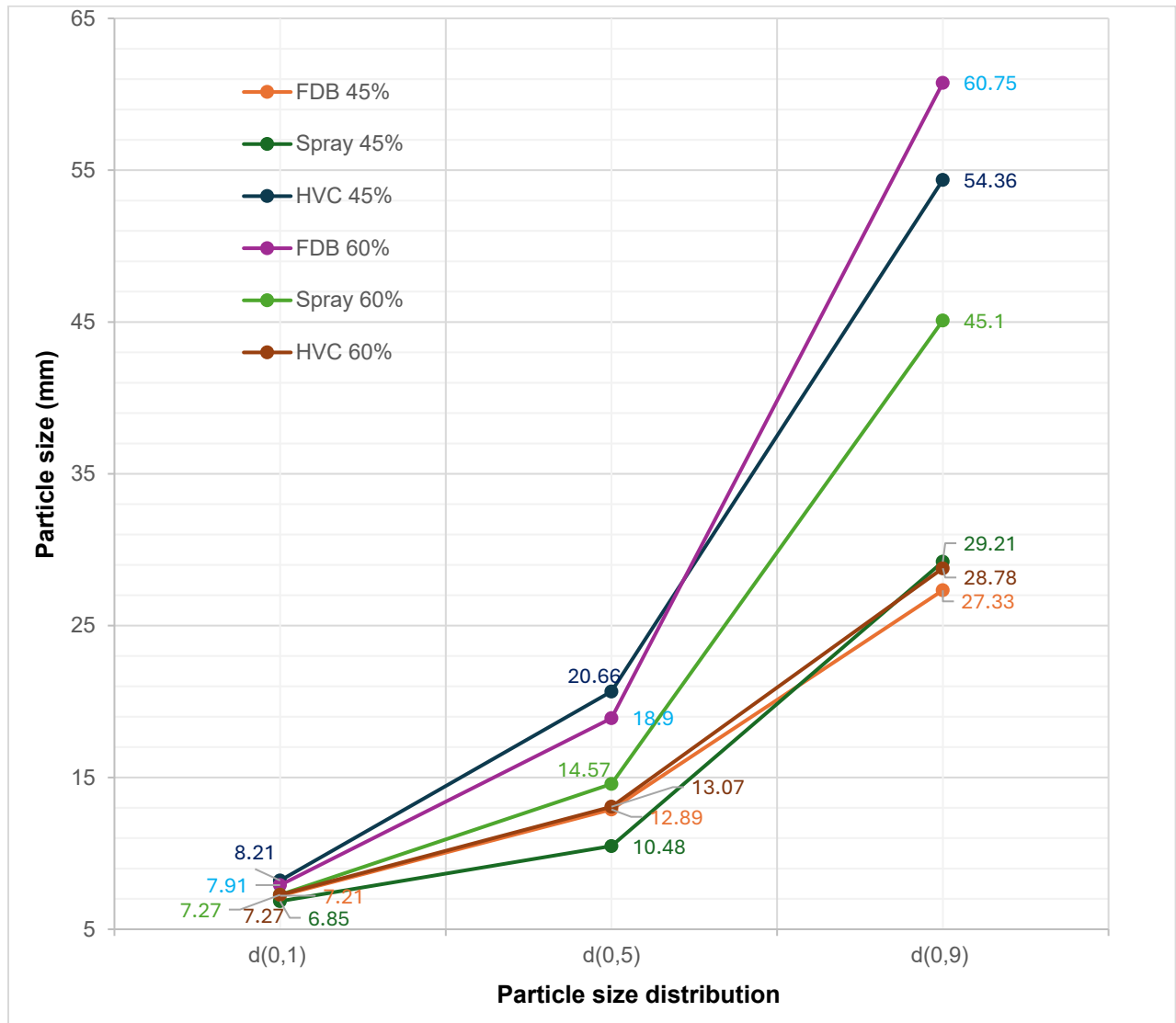


Fig. 6. The particle size distributions $d(0,1)$, $d(0,5)$ and $d(0,9)$ of the samples dried with spray dryer (Spray 45%, Spray 60%), fluidized bed dryer (FDB 45%, FDB 60%) and high-velocity cyclone dryer (HVC 45%, HVC 60%)

Particle Circularity

The circularity results presented in Fig. 7 show that spray drying resulted in the most circular single particles with both raw materials. With the pretreated raw material, RA60%, the circularity of the particles dried with fluidized bed and high velocity cyclone were quite similar. With the never-dried MCC, RA45% as a raw material, the circularity of the particles produced with fluidized bed was quite low compared to the other drying

methods, were the particles obtained from spray and high velocity cyclone dryer showed significant and similar kind of circularity.

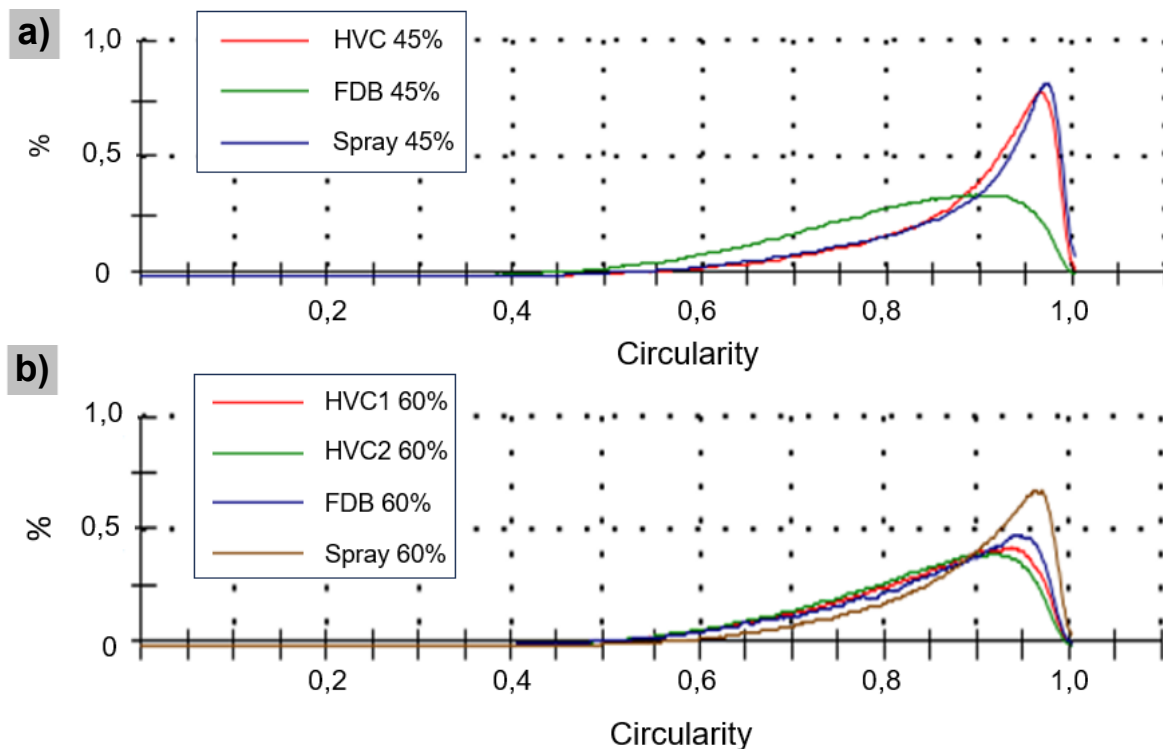


Fig. 7. The particle circularity of the samples dried with spray dryer (Spray 45%, Spray 60%), fluidized bed dryer (FDB 45%, FDB 60%) and high-velocity cyclone dryer (HVC 45%, HVC1 60%, HVC2 60%)

Particle Aggregates

The particle size distribution was problematical to determine accurately due to the irregularity in the particle shapes, particle aggregation, and overlapping among particles. Thus, the shape and sizes of the particle aggregates was examined as well. Figure 8 presents the particle aggregates of the never-dried MCC raw material (dry consistency of 45%), and the samples dried from it with the different techniques. From these results, as well as from the SEM micrographs, it can be noticed how the morphologies of the dried particle aggregates varied between the different drying methods. The raw material MCC consisted of sizeable and highly irregular shapes of crystalline particle aggregates between the sizes of 400 to 600 μm . The aggregates produced with fluidized bed dryer exhibited sizes in the range 200 to 300 μm and of more spherical and smoother appearance than the other samples. The spray dried samples showed much tinier aggregates of particles with a more irregular appearance than the fluidized bed dried ones, the aggregate sizes being in the range 150 to 200 μm . The high velocity cyclone seemed to produce largest aggregates, having sized between 250 to 400 μm , with mixture of irregular, elongated, and diverse particle structures.

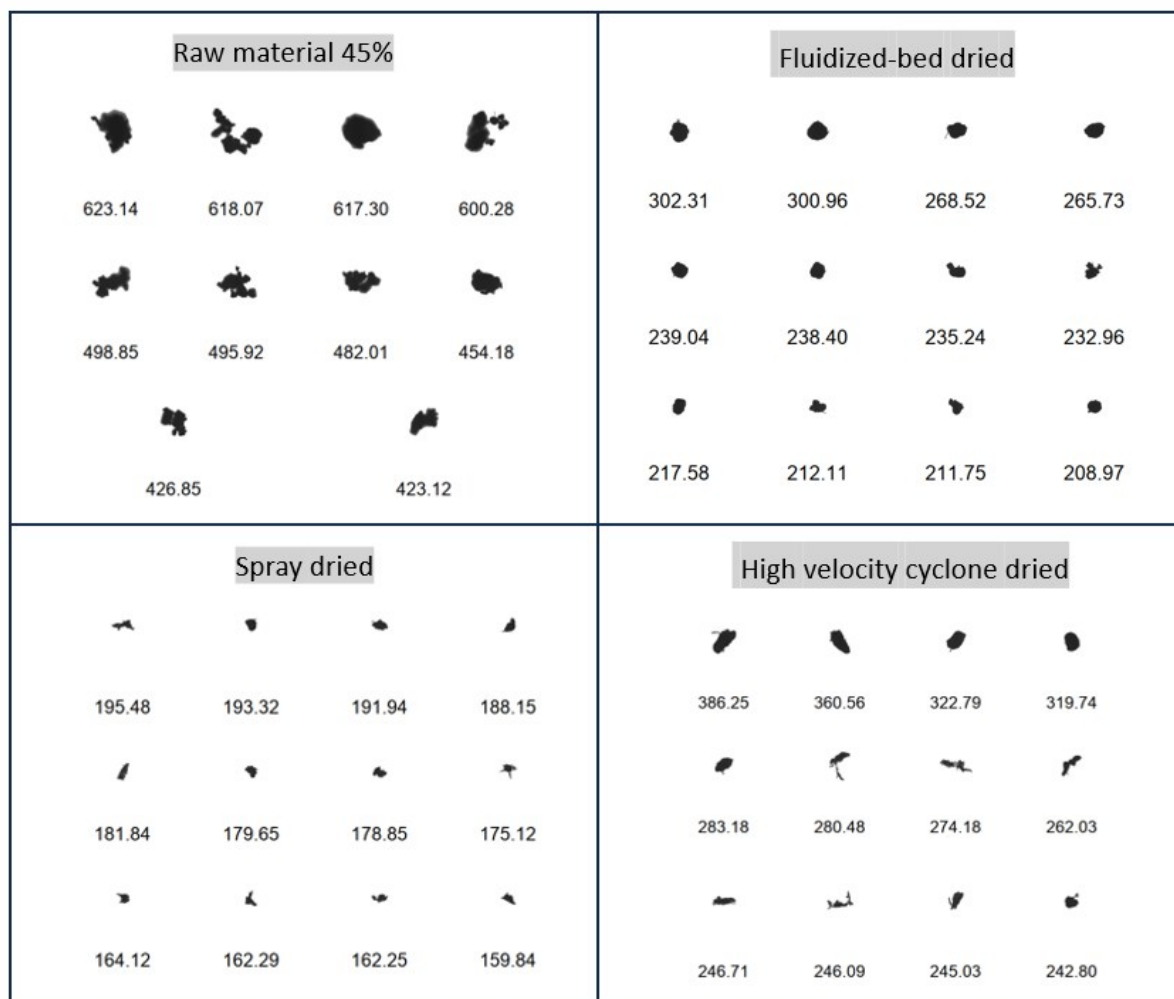


Fig. 8. The shape and size of particle aggregates of the never-dried MCC raw material (dry consistency of 45%), and the samples dried from it with spray dryer, fluidized bed dryer and high-velocity cyclone dryer

Figure 9 presents the particle aggregates of the pretreated MCC raw material (dry consistency of 60%), and the particle aggregate samples dried from it. The pretreated raw material MCC consisted of more spherical aggregate shapes between the sizes of 250 to 500 μm . The aggregates produced with fluidized bed dryer displayed sizes in the range 280 to 350 μm and demonstrated quite similar shapes with its precursor. Additionally, these aggregate samples exhibited more spherical appearance than the samples dried with other methods. Spray dried aggregate samples showed irregular, crystalline shapes with the sizes between 200 and 300 μm . The high velocity cyclone dried aggregates appeared to form irregular shapes from the crystalline particles in the range of 100 to 160 μm .

When comparing the drying results on the particle aggregates based on the raw material MCC, the following observations can be made. The aggregates of the never-dried MCC particles were significantly larger and irregularly shaped than the ones created by the pretreated raw material MCC, since the never-dried MCC had not experienced the hornification phenomena. In general, the fluidized bed aggregates samples dried from the never-dried MCC showed most circular and homogenous shapes and smoothest surface texture of all the dried samples.

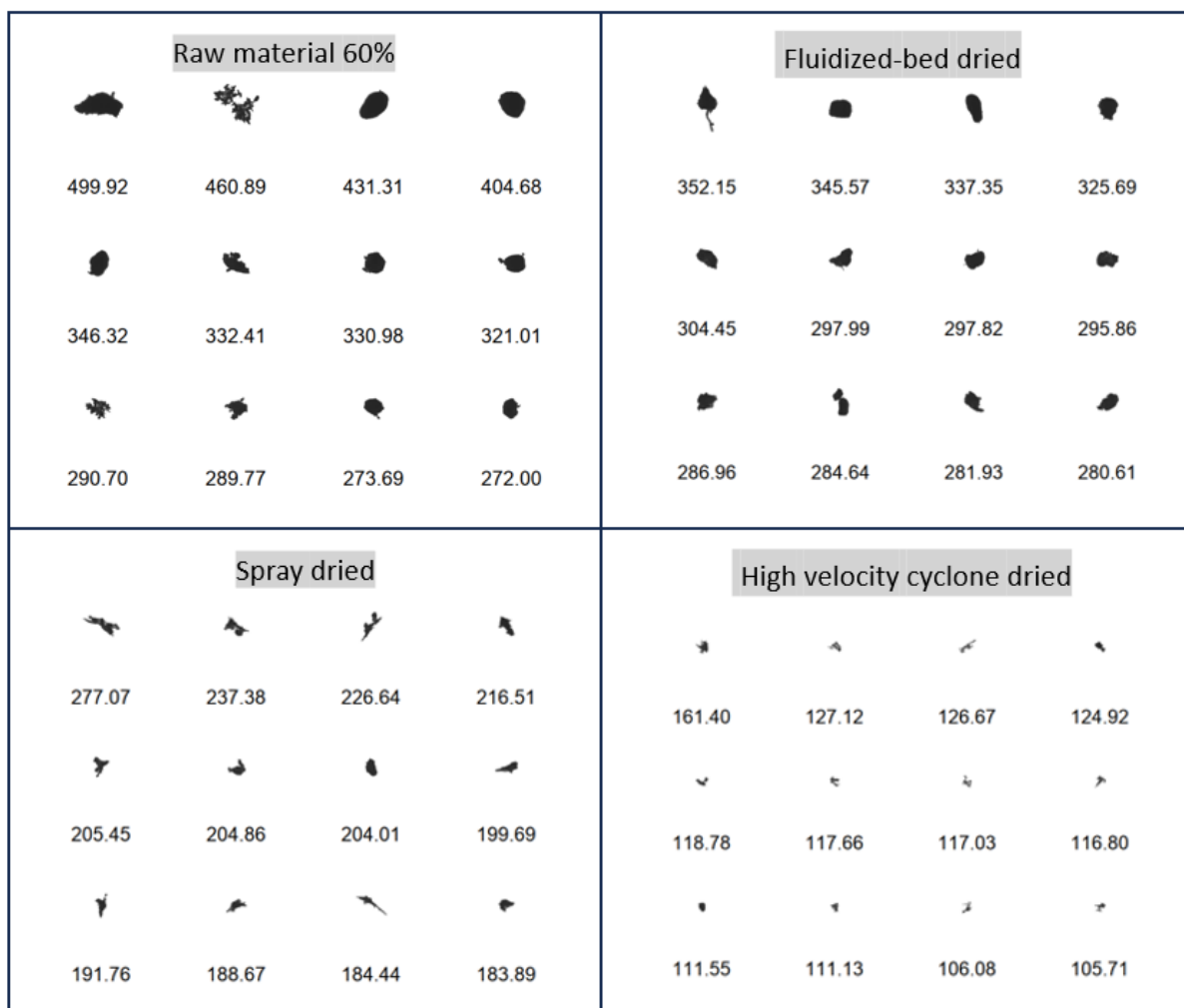


Fig. 9. The shape and size of particle aggregates of the pre-dried MCC raw material (dry consistency of 60%), and the samples dried from it with spray dryer, fluidized bed dryer and high-velocity cyclone dryer

This is most likely due to the extensive hornification happening in the process that closed the particle surfaces during drying and the abrasion inside the device. In addition, the fluidized bed dried aggregates were the largest compared to the other dried samples. The aggregates dried with fluidized bed from the pretreated raw material MCC highly resembled its precursor most likely due to the fact that the greatest part of the hornification already took place in the raw material pretreatment, and no significant hornification occurred any more in the fluidized bed or that the gentler mixing in the fluidized bed was not able to separate the already partly-hornified, rigidly formed particle clusters. That is most likely why the size range of the aggregates from fluidized bed were 100 μm larger with the pretreated MCC compared to the never-dried. Hornification has been found to increase the particle aggregation (Spence *et al.* 2010). This same can be phenomena was also noticed with the spray dried aggregates that were also about 100 μm larger with RA60% as the raw material. Overall, the spray drying produced quite similar forms of aggregates with both raw materials exhibiting crystalline texture and large specific surface area. Only the aggregate samples dried with high velocity cyclone showed more specific surface area and irregular shapes than the spray dried ones. High velocity cyclone dried

aggregate samples demonstrated the greatest differences between the two raw materials, both in sizes and shapes. The size difference between the aggregates dried from the different raw material MCCs was quite significant, from 100 to 300 μm .

CONCLUSIONS

1. The characterization of dried microcrystalline cellulose (MCC) samples confirmed that the drying method as well as the raw material quality significantly influence their physical properties. Each technique produced distinct particle morphologies, even between the different raw materials, indicating the variations in the liquid-MCC interaction and hornification phenomena amongst the drying methods.
2. Scanning electron microscopy (SEM) images indicated that untreated, the never dried MCC raw material presented highly crystalline and irregular surface texture, whereas the pre-dried MCC raw material appeared to have already partially hornified, stiffer fiber particle structure with more closed and smoothened surface structure.
3. Spray drying of the never dried MCC generated tightly packed, spherical, aggregated, and smooth-textured microparticles with reduced surface area, while the pre-dried raw material resulted in more porous, irregular, and crystalline particles and aggregates. Spray drying yielded the most homogeneous particle sizes and significantly reduced particle sizes, especially with the pre-dried MCC.
4. High velocity drying, similar to spray drying, produced larger circular particles with less surface area and aggregation for the untreated MCC and smaller, less aggregated particles for the pre-dried MCC. This method generated the most heterogeneous particle sizes, including the greatest differences in particle sizes and shapes between the two raw materials.
5. The preserving of the raw material crystalline texture was achieved primarily by the fluidized bed, facilitating MCC to dry as discrete particles with high specific surface area and lesser aggregation. Fluidized bed drying produced small, elongated, irregular shape particles with least accumulation and the most circular and homogenous aggregates with smooth textures from the never-dried raw material, while the pre-dried material mostly retained their precursor's characteristics.

ACKNOWLEDGMENTS

The authors are grateful for all the support given by the staff of the Department of Bioproducts and Biosystems, Aalto University School of Chemical Technology.

REFERENCES CITED

- Amin, M. C. I. M., Abadi, A. G., and Katas, H. (2014). "Purification, characterization and comparative studies of spray-dried bacterial cellulose microparticles," *Carbohydrate Polymers* 99, 180-189. DOI: 10.1016/j.carbpol.2013.08.04
- Balaxi, M., Nikolakakis, I., Kachrimanis, K., and Malamataris S. (2009). "Combined effects of wetting, drying, and microcrystalline cellulose type on the mechanical strength and disintegration of pellets," *Journal of Pharmaceutical Science* 98, 676-689. DOI: 10.1002/jps.21454
- Battista, O. A., and Smith, P. A. (1962). "Microcrystalline cellulose – The oldest polymer finds new industrial uses," *Industrial and Engineering Chemistry Research* 54, 20-29.
- Beck, S., Bouchard, J., and Berry, R. (2012). "Dispersibility in water of dried nanocrystalline cellulose," *Biomacromolecules* 13(5), 1486-1494. DOI: 10.1021/bm300191k
- Cal, K., and Sollohub, K. (2010). "Spray drying technique. I: Hardware and process parameters," *Journal of Pharmaceutical Sciences* 99(2), 575-586. DOI: 10.1002/jps.21886
- Daud, W. R. W. (2008). "Fluidized bed dryers – Recent advances," *Advanced Powder Technology* 19, 403-418. DOI: 10.1016/S0921-8831(08)60909-7
- Dehdarinejad, E., and Bayareh, M. (2023). "Analysis of the vortical flow in a cyclone using four vortex identification methods," *Powder Technology* 428, article 118897. DOI: 10.1016/j.powtec.2023.118897
- Diniz, J. M. B. F., Gil, M. H. J. A., and Castro, A. M. (2004). "Hornification its origin and interpretation in wood pulps," *Wood Science Technology* 37, 489-494. DOI: 10.1007/s00226-003-0216-2
- FMC (2015). "Avicel for solid dose forms - FMC BioPolymer," (<http://www.fmcbiopolymer.com/Pharmaceutical/Products/Avicelforsoliddoseforms.aspx>), Accessed January 10, 2025.
- Grimm, A., Elustondo, D., Mäkelä, M., Segerström, M., Kalén, G., Fraikin, L., Léonard, A., and Larsson, S. (2017). "Drying recycled fiber rejects in a bench-scale cyclone: Influence of device geometry and operational parameters on drying mechanisms," *Fuel Processing Technology* 167, 631-640. DOI: 10.1016/j.fuproc.2017.08.004
- Haafiz, M. K. M., Hassan, A., Zakari, Z., and Inuwa, I. M. (2014). "Isolation and characterization of cellulose nanowhiskers from oil palm biomass microcrystalline cellulose," *Carbohydrate Polymers* 103, 119-125. DOI: 10.1016/j.carbpol.2013.11.055
- Hovmand, S. (2020). "Fluidized bed drying," in: *Handbook of Industrial Drying*, A.S. Mujumdar (ed.), CRC Press, Boca Raton, FL, USA.
- Ioelovich, M. (2019). "Study of fractal dimensions of microcrystalline cellulose obtained by the spray-drying method," *Fractal and Fractional* 3(1), article 3. DOI: 10.3390/fractalfract3010003
- JRS (2015). "EMCOCEL® and VIVAPUR® - Microcrystalline Cellulose - JRS Pharma. (http://www.jrspharma.com/pharma_en/products-services/excipients/binders/emcocel-vivapur.php), Accessed January 10, 2025.
- Kamel, S., Jahangir, N. A. K., Shah, S. M., and El-Gendy, A. A. (2008). "Pharmaceutical significance of cellulose: A review," *Express Polymer Letters* 2(11), 758-778. DOI: 10.3144/expresspolymlett.2008.90
- Krawczyk, G., Venables, A., and Tuason, D. (2009). "Microcrystalline cellulose," in:

- Handbook of Hydrocolloids*, G. O. Phillips, and P. A. Williams (eds.), Woodhead Publishing, Cambridge, UK.
- Liu, Y., Peng, Y., Kansha, Y., Ishizuka, M., Tsutsumi, A., Jia, D., Bi, X. T., Lim, C. J., and Sokhansanj, S. (2014). "Novel fluidized bed dryer for biomass drying," *Fuel Processing Technology* 122, 170-175. DOI: 10.1016/j.fuproc.2014.01.036
- Mills, S. (2007). "Pharmaceutical excipients – An overview including considerations for paediatric dosing," in: *World Health Organization, Training Workshop on Pharmaceutical Development with focus on Paediatric Formulations*, Tallinn, Estonia.
- Mujumdar, A. S. and Devahasthin, S. (2003). "Applications of fluidized bed drying," in: *Handbook of Fluidization and Fluid Systems*, W.C. Yang (ed.), Marcel Dekker, New York, NY.
- Nebra, S. A., Silva, M. A., and Mujumdar, A. S. (2000). "Drying in cyclones - A review," *Drying Technology* 18, 791-832. DOI: 10.1080/07373930008917738
- Nwachukwu, N., and Ofoefule, S. I. (2017). "Effect of drying methods on the powder and compaction properties of microcrystalline cellulose obtained from *Cocos nucifera*," *Journal of Pharmaceutical Research International* 20(2), 1-15. DOI: 10.9734/JPRI/2017/37615
- Nwachukwu, N., and Ofoefule, S. I. (2020). "Effect of drying methods on the powder and compaction properties of microcrystalline cellulose derived from *Gossypium herbaceum*," *Brazilian Journal of Pharmaceutical Sciences* 56(1), article 118060. DOI: 10.1590/s2175-97902020000118060
- Oliveira, O. W., and Petrovick, P. R. (2009). "Spray drying (spray drying) of plant extracts: Basic remarks and applications," *Pharmacognosy Journal* 20, 641-650.
- Pandey, J. K., Takagi, H., and Nakagaito, A. N. (2012). "An overview on the cellulose based conducting composites," *Composites Part B: Engineering* 43, 2822-2826. DOI: 10.1016/j.compositesb.2012.04.045
- Paudel, A., Worku, Z. A., Meeus, J., Guns, S., and Van den Mooter, G. (2013). "Manufacturing of solid dispersions of poorly water soluble drugs by spray drying: Formulation and process considerations," *International Journal of Pharmaceutics* 453(1), 253-284. DOI: 10.1016/j.ijpharm.2012.07.015
- Peng, Y., Gardner, D. J., and Han, Y. (2012). "Drying cellulose nanofibrils: In search of a suitable method," *Cellulose* 19, 91-102. DOI: 10.1007/s10570-011-9630-z
- Peng, Y., Han, Y., and Gardner, D. J. (2012). "Spray-drying cellulose nanofibrils: Effect of drying process parameters on particle morphology and size distribution," *Wood and Fiber Science* 44 (4), 448-461.
- Spence, K. L., Venditti, R. A., Rojas, O. J., Habibi, Y., and Pawlak, J. J. (2010). "The effect of chemical composition on microfibrillar cellulose films from wood pulps: Water interactions and physical properties for packaging applications," *Cellulose* 17, 835-848. DOI: 10.1007/s10570-010-9424-8
- Sjöstrand, B., Karlsson, C.-A., Barbier, C., and Henriksson, G. (2023). "Hornification in commercial chemical pulps: Dependence on water removal and hornification mechanisms," *BioResources* 18(2), 3856-3869. DOI: 10.15376/biores.18.2.3856-3869
- Thoorens, G., Krier, F., Leclercq, B., Carlin, B., and Evrard, B. (2014). "Microcrystalline cellulose, a direct compression binder in a quality by design environment—A review," *International Journal of Pharmaceutics* 473(1-2), 64-72. DOI: 10.1016/j.ijpharm.2014.06.055
- Transparency market research. (2015). "Microcrystalline cellulose (MCC) market for

- food and beverage, pharmaceutical, personal care and other end-users - Global industry analysis, size, share, growth, trends and forecast 2014 – 2020,” Transparency Market Research, Albany, United States.
- Tuason, D. C., Krawczyk, C. R., and Buliga, G. (2012). “Microcrystalline cellulose,” in: *Food Stabilisers, Thickeners and Gelling Agents*, A. Imeson (ed.), Wiley-Blackwell, Chichester, UK.
- Vanhatalo, K. (2017). *A New Manufacturing Process for Microcrystalline Cellulose (MCC)*, Ph.D. Dissertation, Aalto University, Espoo, Finland.
- Vanhatalo, K., and Dahl, O. (2014). ”Effect of mild acid hydrolysis parameters on properties of microcrystalline cellulose,” *BioResources* 9, 4729-4740. DOI: 10.15376/biores.9.3.4729-4740
- Vasiliu-Oprea, C., and Nicoleanu, J. (1993). “Micronized (and microcrystalline) celluloses. Obtainment and fields of application,” *Polymer-Plastics Technology and Engineering* 32, 181-214. DOI: 10.1080/03602559308019227
- Walton, D., and Mumford, C. (1999). “Spray dried products—Characterization of particle morphology,” *Chemical Engineering Research and Design* 77(1), 21-38.
- Zimmermann, M., Borsoi, C., Lavoratti, A., Zanini, M., Zattera, A., and Santana, R. (2016). “Drying techniques applied to cellulose nanofibers,” *Journal of Reinforced Plastics and Composites* 35. DOI: 10.1177/0731684415626286

Article submitted: February 10, 2025; Peer review completed: April 5, 2025; Revised version received: May 5, 2025; Accepted: June 18, 2025; Published: September 9, 2025. DOI: 10.15376/biores.20.4.9524-9541