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Frédéric Courteille, Florent Bourgeois, Michaël Clifton, Martine Meireles. 3D Reconstruction of Ultrafiltration Cakes from Binarised Images. FILTECH 2009, International Conference & Exhibition for Filtration and Separation Technology, Oct 2009, Wiesbaden, Germany. pp.0. hal-04104914

HAL Id: hal-04104914

<https://hal.science/hal-04104914>

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To cite this version :

Courteille, Frédéric and Bourgeois, Florent and Clifton, Michaël and Meireles, Martine (2009) *3D Reconstruction of Ultrafiltration Cakes from Binarised Images*. In: FILTECH 2009, International Conference & Exhibition for Filtration and Separation Technology, 13-15 octobre 2009, Wiesbaden, Allemagne.

3D RECONSTRUCTION OF ULTRAFILTRATION CAKES FROM BINARISED IMAGES

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ABSTRACT

Transmission electron microscopy (TEM) imaging is a common technique for studying microstructure of ultrafiltration cakes. It yields local microstructural information in the form of two-dimensional gray-level images of slices a few particle diameters in thickness. This work presents a simple yet powerful particle-by-particle reconstruction scheme for simulating ultrafiltration cake microstructure from TEM images. The scheme uses binarised TEM images, thereby permitting use of lesser quality TEM images. Moreover, the reconstruction scheme embeds the multi-scale nature of ultrafiltration cake microstructure using a novel concept that consists in matching the morphology of simulated and measured structures at a number of scales identifiable in the microstructure. In the end, reconstructed microstructures are intended for bettering our understanding of the relationships between cake morphology, ultrafiltration performance and operating conditions.

KEYWORDS

Ultrafiltration, Cake microstructure, 3D reconstruction

1. Problem statement

Water purification using ultrafiltration membranes is an industrial-scale process that replaces both coagulation and sand filtration. At present, stakes are high for increasing the cost-effectiveness of the process. One key dimension to this problem is the correlation between ultrafiltration efficiency (production rate and filtrate quality) and the microstructure of the cake that forms on the membrane surface during the process. As in other filtration processes, cake microstructure is a crucial element in this problem. But ultrafiltration differs from other filtration processes in that direct observation of the morphology of an ultrafiltration cake is particularly difficult. TEM images are used to capture local information about ultrafiltration cake microstructure. The gray-level information of TEM images is potentially valuable for spatial reconstruction. However, it turns out that the quality of TEM images, when imaging nano-structures is often insufficient for making quantitative use of the gray-level information. Hence, there is value in developing a reconstruction scheme that uses binarised TEM images only. Such images correspond to the projection of the particles captured by TEM images onto a plane situated anywhere inside the cake slice captured by the TEM scan. A typical TEM image, with its binarised transform is shown in Fig. 1 for an ultrafiltration cake obtained using 26 nm monodispersed spherical particles. The thickness of the cake slice captured in this TEM image is 80 nm, which is typical of what can be expected from current TEM machines.

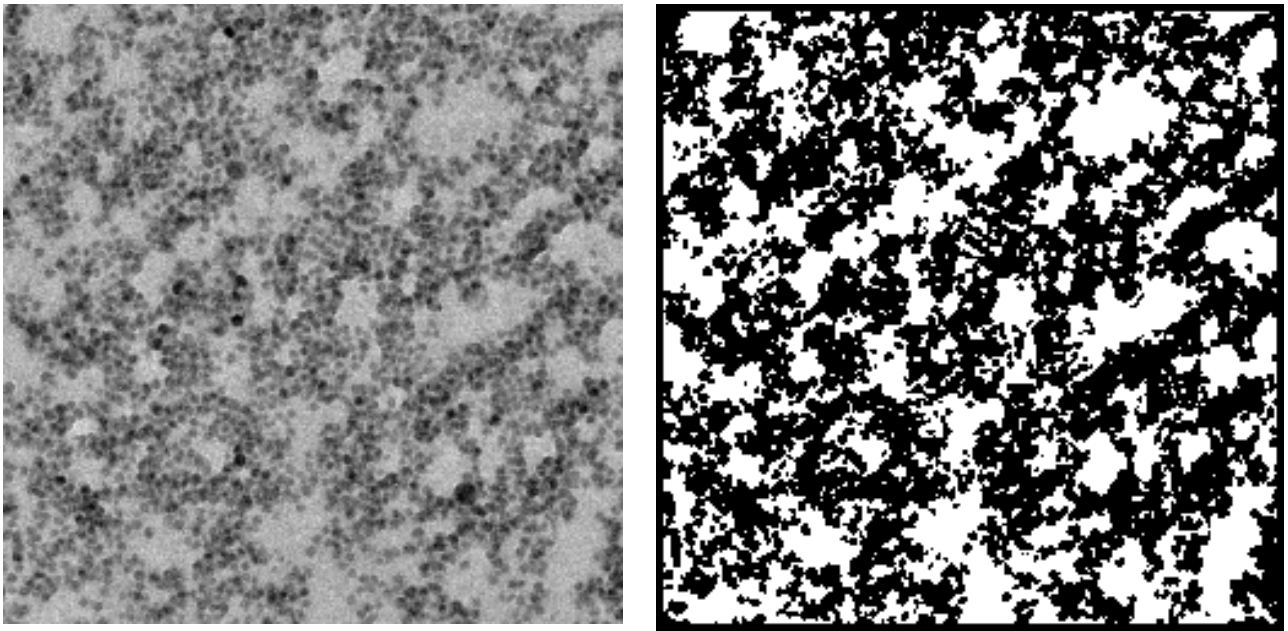


Figure 1. Original and binarised TEM image of an ultrafiltration cake (image size $\approx 1 \mu\text{m}^2$)

2. 3D reconstruction scheme

In order to reconstruct ultrafiltration cakes from cross-section images, several options can be selected. At one end of the spectrum of reconstruction solutions, one finds statistical models such as the Cox process used by Jeulin and Moreau (2005). With such an approach, Jeulin and Moreau were able to extract the parameters of several Boolean processes, which they combined to reconstruct microstructures with aggregates. In general, statistical models can be used provided particles are arranged according to some process whose spatial statistical properties can be extracted from morphological functions measured on 2D images. This approach is elegant and attractive both computationally and analytically. At the other end of the spectrum, one may consider reconstruction schemes which we shall refer to as global. For example, Oberdisse et al. (2007) have used the reverse Monte-Carlo (RMC) technique for simulating fractal aggregates. Although they did not start from images, as they used small-angle neutron scattering (SANS) information instead, their procedure starts from a randomly formed aggregate, which they rearrange using the RMC algorithm until the SANS spectrum of the reconstructed aggregate matches the measured one. In principle, global techniques can be applied to structures larger than single aggregates; however the task becomes more complex as the number of particles increases. Moreover, choosing the number of unknowns is a critical step with such reconstruction schemes, as the reconstructed volume must be filled with particles before the start of the rearrangement process. The particle rearrangement algorithm is computer intensive; however there is no reason why such an approach could not be made to work satisfactorily.

The procedure developed in this paper sits somewhere in between purely statistical and global approaches. The former was not selected on the basis that there is no evidence that ultrafiltration cakes conform to any particular statistical configuration. The authors avoided using the latter approach because of foreseeable difficulties with simulating large volumes. The proposed reconstruction scheme is an incremental particle-by-particle reconstruction of the cake slice that corresponds to the volume captured by the TEM image. Adding particles one by one avoids having to specify the number of particles *a priori* and circumvents the difficult choice of this unknown. At every iteration, i.e. every time a new particle is added into the system, the point $P(x,y,z)$ at which it is implanted is chosen such that it minimizes the difference between the specific surface area of the real microstructure

and the specific surface area of the reconstructed microstructure at the current iteration with the new particle added at point P . In practice, the new particle can be positioned at random locations of two types. If the targeted specific surface area is less than that measured at the end of the previous particle addition step (see Fig. 2), the particle is positioned at a randomly chosen location such that its projected area does not overlap with that of other particles; hence it contributes its whole perimeter to the specific surface area. Otherwise, as shown in Fig. 3, it is positioned at a randomly chosen location such that its projected area overlaps with that of other particles, thereby contributing only marginally to the change in specific surface area.

The specific surface area can be calculated directly from the image by counting pixels that are located at the interface between the porous and solid phases. However, the function that is used in this work is the classical two-point covariance function S (Serra, 1982), which is defined over the set of chords of length h . Not only S is a reliable means for calculating specific surface area from binary images (Berryman, 1985), but it also captures a fine morphological signature of the texture. The authors have found that, despite the fact that the proposed reconstruction algorithm relies on matching the specific surface area only, the two-point covariance function of the textures reconstructed with the proposed method matches that of the original textures over significant chord lengths. The reason for this interesting finding is believed to be the combination of the multi-scale reconstruction scheme that is presented later, and the specified particle shape, which constrains the covariance function significantly.

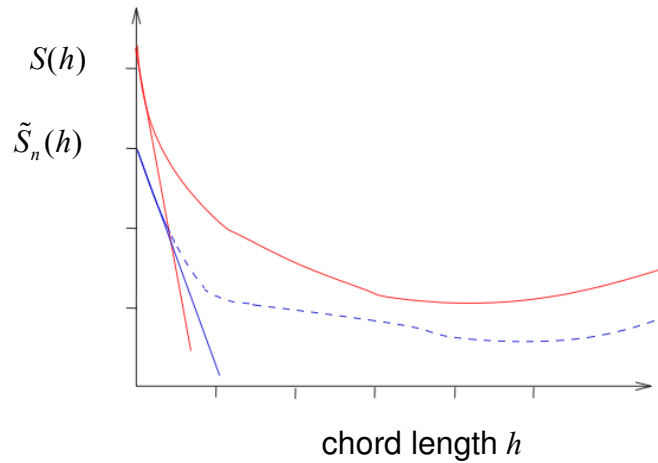
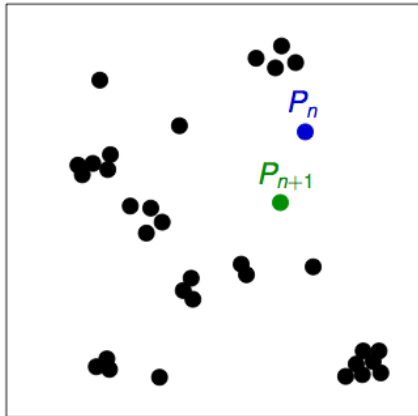


Figure 2. The targeted specific surface area is less than that measured at iteration n , hence particle P_{n+1} is positioned at iteration $n+1$ at random inside the reconstructed volume such that its projected area does not overlap with that of other particles. S and \tilde{S}_n correspond to the two-point covariance function of the targeted image (solid line) and the reconstructed structure (dotted line) after n iterations respectively.

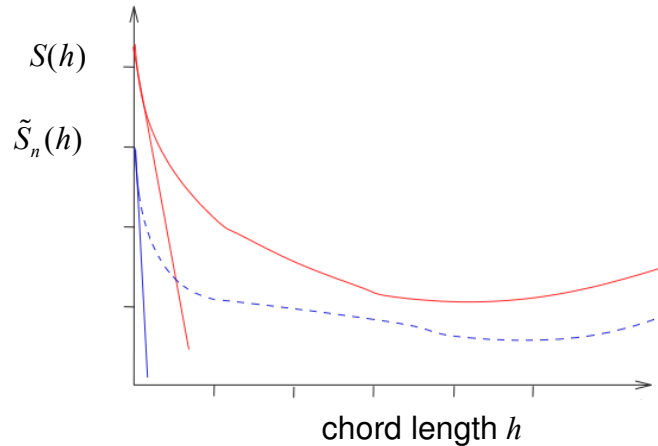
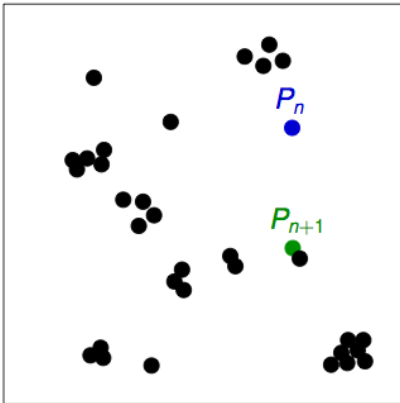


Figure 3. The targeted specific surface area is higher than that measured at iteration n , hence particle P_{n+1} is positioned at iteration $n+1$ at a position such that its projected area overlaps with that of other particles. S and \tilde{S}_n correspond to the two-point covariance function of the targeted image (solid line) and the reconstructed structure (dotted line) after n iterations respectively.

The next issue consists in capturing the multi-scale nature of the texture from the image of the texture, from single particles to large aggregates. Quantification of the multi-scale information of the texture is achieved in two successive steps, as described in Fig. 4: step one, a set of images of individual scales is generated by successive application of an erosion operator¹; step two, the 2-point covariance function is computed for each scale.

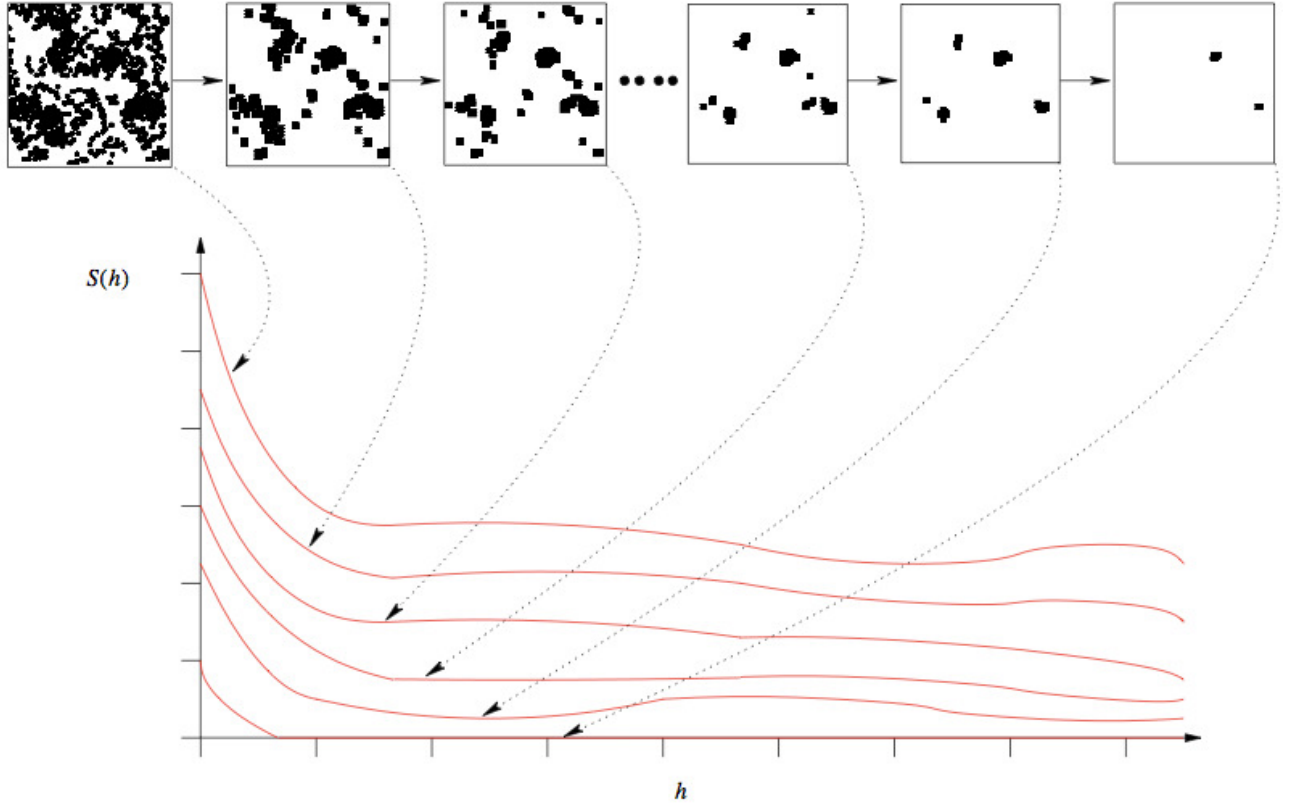


Figure 4. Illustration of the multi-scale image analysis scheme.

The multi-scale texture simulation algorithm then simply consists in matching the two-point covariance function at every individual scale, starting from the upper scale (rightmost image in Fig. 4) and moving to the left of Fig. 4 towards the lower scales. Addition of particles reduces the porosity, hence the two-point covariance function $S(h)$ moves upward every time a particle is added to the system. Once the porosity of the current scale is matched, the algorithm uses the slope of the two-point covariance function of the next scale as the next target for the particle-by-particle addition scheme presented earlier.

3. Results and analysis

Firstly, the simulation algorithm was tested against images obtained from slices through an artificial cake generated using the physico-chemical model developed by Madeline et al.

¹ Erosion is one of the two elementary operators in the area of mathematical morphology, the other being dilation. The basic effect of the operator is to erode away the boundaries of regions of foreground pixels (black pixels).

(2007). This is an ideal situation, in that particles in the images are spherical and every spatial property is known for the particles inside the cake, making it possible to develop and fully test the validity of the reconstruction scheme.

Fig. 5 shows the results obtained when simulating with the proposed algorithm the texture whose frame is bolded. Fig. 5 was deliberately presented as a children's "Spot the difference" game, with the original cake slice image placed amongst 8 simulations. Should the bold frame be removed, it then becomes rather difficult to spot the original image in Fig. 5, thereby confirming the potential of the proposed 3D simulation technique for ultrafiltration cake slice simulation. We note however some differences with the reconstruction of the aggregates. The cause is an insufficient sampling of the larger particle aggregates in the original image, due to the fact that the technique used to generate the artificial ultrafiltration cake could only produce small cake volumes.

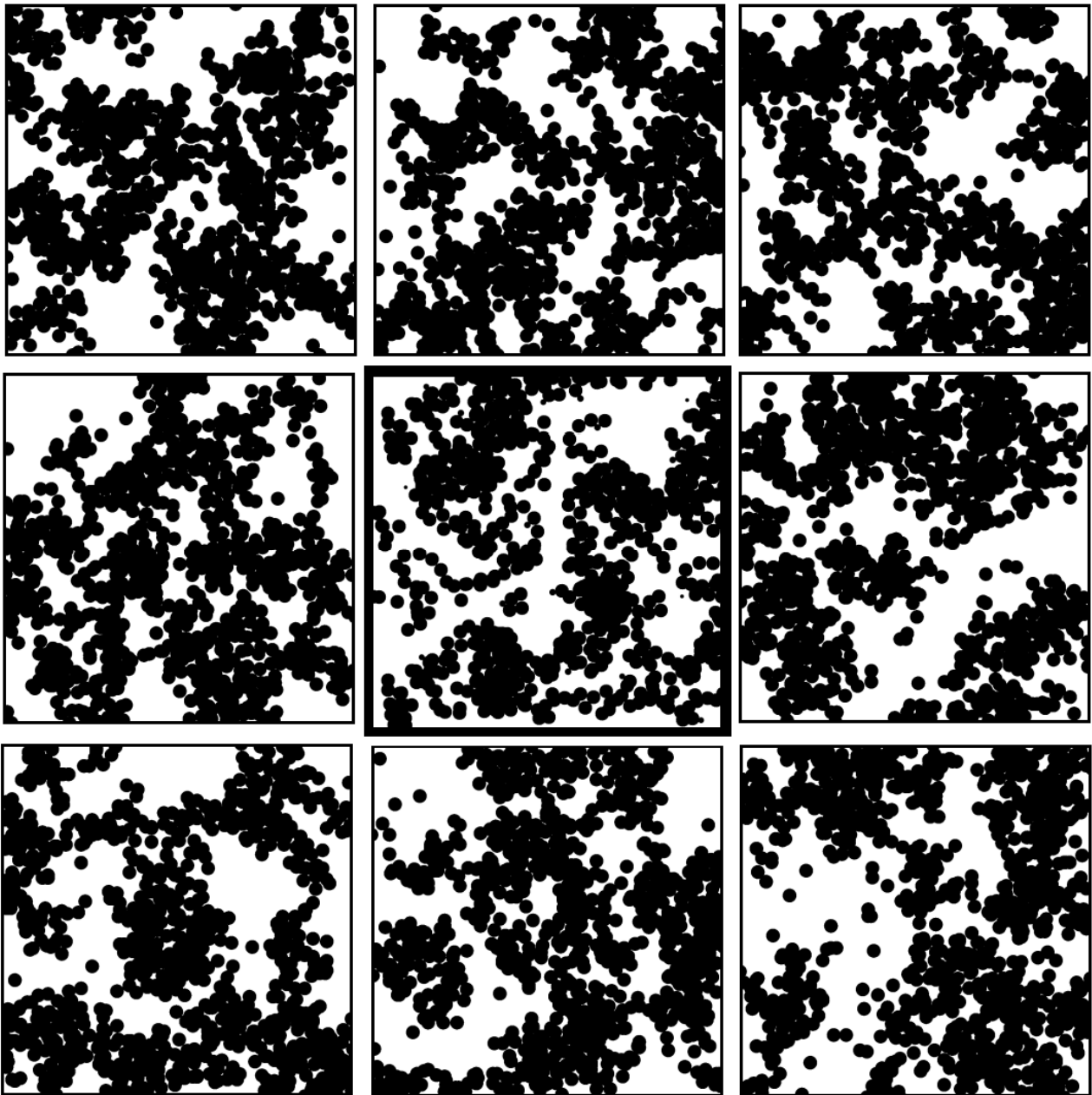


Figure 5. Projected image of the artificial ultrafiltration cake slice (bold frame) and 8 projected images of simulated slices using the proposed reconstruction method.

The second application of the proposed method is the reconstruction of the binarised TEM image of a real ultrafiltration cake, which was presented in Fig. 1. Contrary to the artificial cake reconstructed in Fig. 5, the sampling of the aggregates is quite satisfactory, as the field of view is large relative to the size of the largest aggregates. One reconstruction result is shown in Fig. 6, directly to the right of the original image. A notable difference with the previous ideal example is that particles no longer appear as spheres in the raw image. This is due to both the size of the particles (26 nm nominal diameter) and the binarisation step applied to the gray-scale TEM image. Nevertheless, the projected image of the simulated slice gives a satisfactory match to the original image. Moreover, as we hinted earlier, Fig. 6 confirms the match between the simulated and measured 2-point covariance function for a number of intermediate scales.

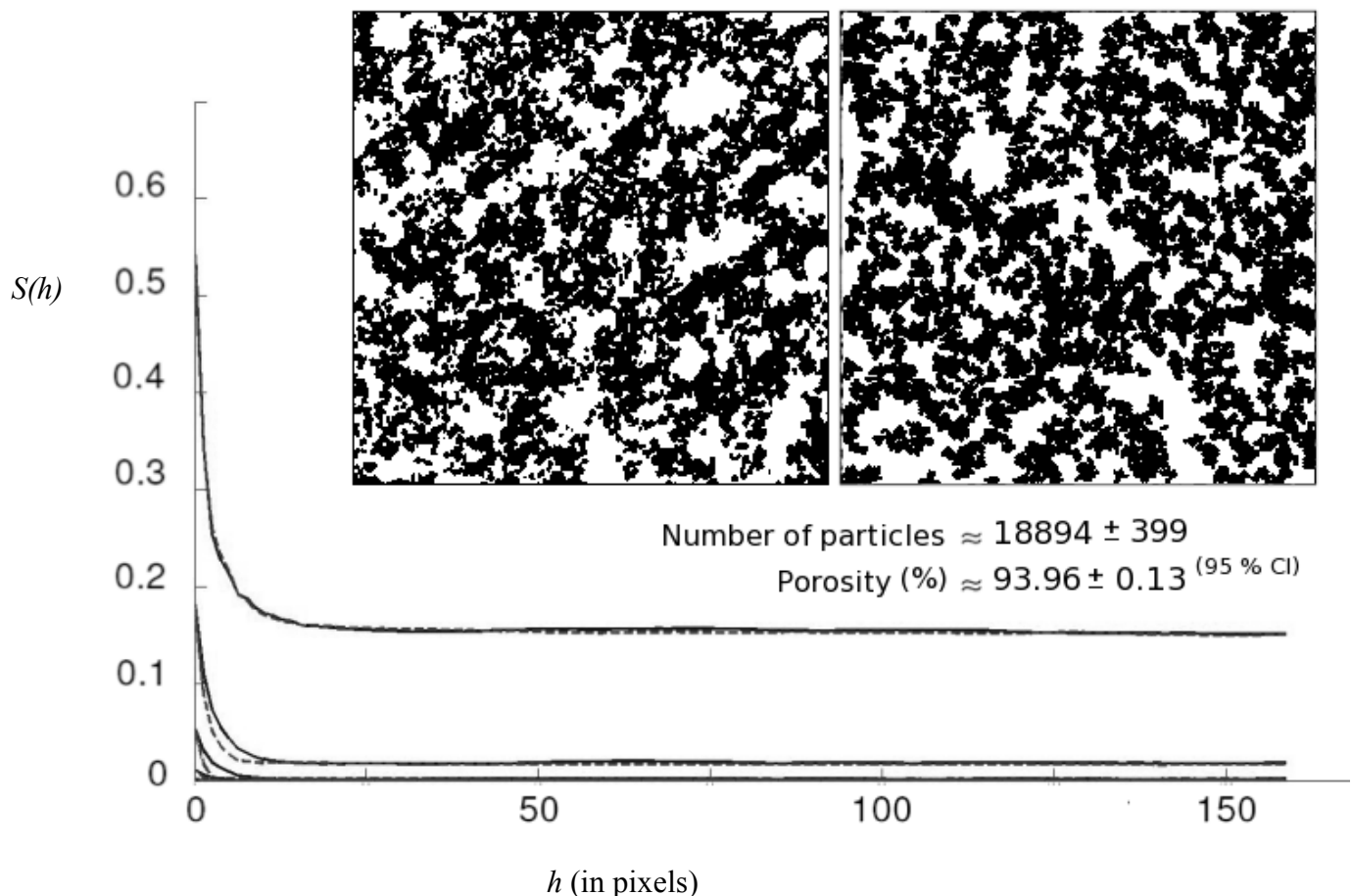


Figure 6. 3D simulation of a real ultrafiltration cake slice (original binarised TEM image to the left; simulated image to the right). Measured 2-point covariance functions are represented as a solid line, whereas simulated ones are shown as dotted lines. Confidence intervals are given for 300 simulations.

Visually, all reconstructed images are satisfying. One observes that the simulation scheme recovers correctly both the void space and the state of particle aggregation. The state of aggregation is better recovered than with the artificial cake used earlier as the raw image provides a good sampling of the morphological features that correlate over long distances. Quantitatively, the quality of the convergence of the distance watershed objective function can be assessed using chi-square statistics, which shows equality between measured and reconstructed objective functions with a confidence greater than 95%. In the end, the interfacial area is precisely matched, as confirmed by the match between the slopes of the 2-point covariance functions near the origin. Some improvement to the particle-by-particle addition scheme could be made in order to match the covariance functions over a longer range of chord length h . Indeed, because particles of given shape and size distribution are

added into the system, the shape of the resulting covariance function is strongly constrained. This explains why the simple particle-by-particle addition method embedded into the proposed method yields in the end a good match of the measured 2-point covariance function even at relatively long chords. The reader should also notice that the simulated covariance function is not expected to match the measured one at long chord lengths since the uncertainty of the measurements is directly proportional to the chord length h . Hence, the authors believe that a further improvement would be required should the technique be applied to textures whose scales cover several orders of magnitude.

Fig. 7 gives a 3D view of one of the cake slices whose projection was shown in Fig. 5. The reconstruction scheme described here uses binarised TEM images only, which do not contain information that can be used directly to derive particle position in the vertical direction. Without additional constraint, the reconstruction algorithm yields a particle network that is looser than the real one. This problem is successfully overcome by using the abovedescribed algorithm to position particles in the X-Y plane, and then selecting vertical positions that favour inter-particle contact in 3D when possible (i.e. when the particle's projection is not isolated in the projected image of the slice under reconstruction). This constraint embeds the physics of particle deposition in an ultrafiltration cake, which dictates that particles must form a connected network. The authors have checked from artificial cakes (See earlier example illustrated in Fig. 5) that the number of particles that are implanted in the reconstructed volume is very close to the number of particles inside the original cake slice. One explanation is that ultrafiltration cakes are very dilute systems, so almost every particle inside a slice a few particle diameters in thickness will contribute something to the projected binary image of that slice. In other words, only very few particles can possibly hide directly behind others inside the thickness of a cake slice captured by TEM. Nevertheless, the reconstruction scheme presented in this paper does not yield the exact number of particles inside the slice, nor can it possibly decide what the best vertical position might be for each particle.

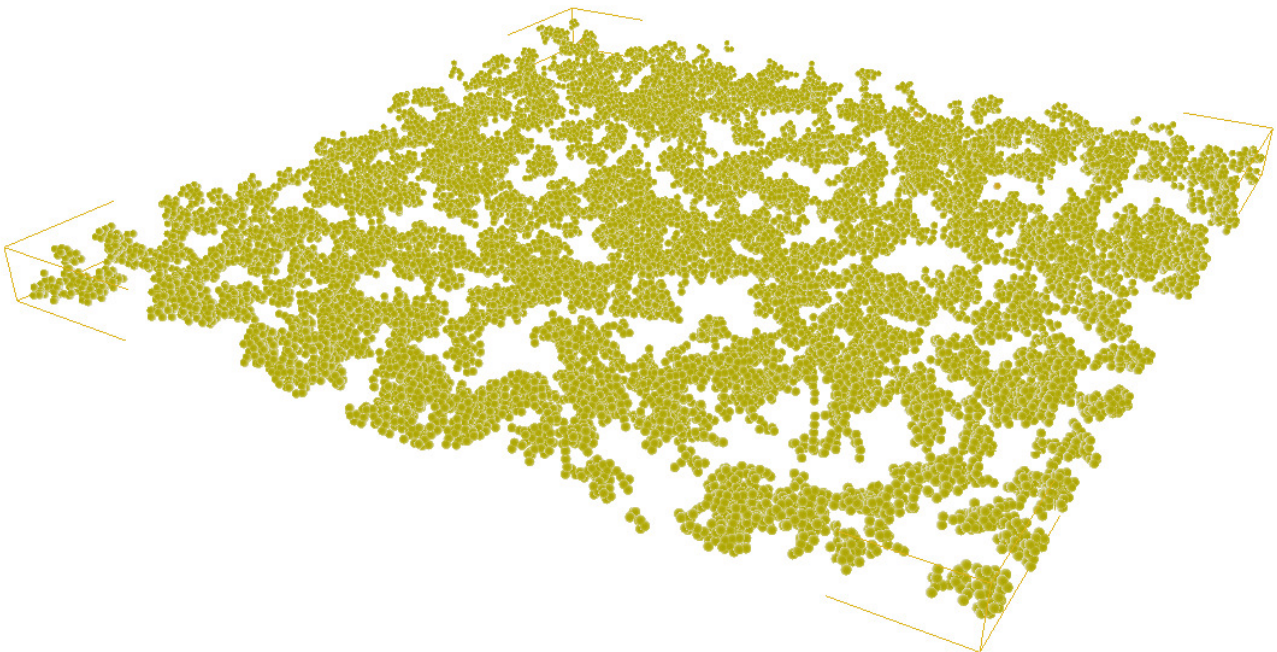


Figure 7. Example of a 3D ultrafiltration cake slice simulated with the proposed method.

Before concluding, it should be stressed that a major advantage of reconstructing 3D textures is that it permits generation of many different random realisations, thereby providing confidence intervals for any property that can be derived from microstructure simulations. This is a particularly important practical point as it is often the case that only one single ultrafiltration cake image is available for a given set of experimental conditions.

4. Conclusions and perspectives

This article has presented a new scheme for simulating ultrafiltration cakes from binarised TEM images. Such images can be obtained routinely in practice even from poor quality gray-level images: this makes the approach particularly valuable for microstructure studies of nanostructures.

The originality of the proposed 3D simulation scheme is twofold. Firstly, the scheme uses a particle-by-particle addition process. Hence, the 3D simulation does not rely upon any assumption about the spatial statistics of the system, and simulated volumes are not limited in size as with global numerical reconstruction schemes. Secondly, the proposed simulation scheme simulates multi-scale textures successfully by matching the morphology of simulated and measured textures at every individual scale. The morphological properties of the texture at every scale are quantified using the 2-point covariance function. Because the scheme adds particles of given shape and size distribution into the reconstruction volume, it is found that it is sufficient to match the y-intercept and the slope near the origin of the 2-point covariance function in order to obtain satisfactory 3D simulations. Hence, the proposed technique is quite straightforward to implement. Convincing simulation results were obtained with both ideal and real ultrafiltration cakes.

In a forthcoming publication, the authors will present a generalized version of this 3D simulation algorithm that solves the problem of reconstructing a whole cake from one or more TEM images. The principle consists in using a moving reconstruction volume whose thickness equals that of the TEM image, centred on the vertical position of the particle that is added during the particle-by-particle addition reconstruction scheme. Hence, a volume larger than the simulated slice is built in the end, in such a way that every slice centred on every particle inside the system has a projected image whose 2-point covariance function matches the measured values. Using an elevation-invariant covariance function, or an elevation variant one, the technique is able to reconstruct not just one single cake slice, but a whole ultrafiltration cake. Moreover, the technique that is developed here with spheres can readily be applied using other particle shapes (e.g. polyhedra) and distributions of particle size. Hence, with such a technique, which is quite simple to implement, one is able to simulate a wide variety of multi-scale two-phase materials. It is worth noting that the 3D simulation technique presented here is also extendable to multi-phase systems, using inter-phase 2-point covariance functions.

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