Pore Size Estimation

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Abstract: - A novel method for characterization of any porous structure is presented. The principle of the method is simple scanning a plane surface of a cellular material and consequent digital image analysis of the scan. Eleven samples of bread baked from dough with different ratios wheat/rye flour were studied in order to determine the volume of pores and pores volume distribution in the samples. The procedure included drying the samples and scanning their surfaces finely covered by stamp ink. The obtained images of contrast-visualized pore boundaries were converted to the black and white form and computationally analyzed using the principles of Euclidean distance map and Voronoi tessellation. The computed pore area sections were compared with a model set of data based on generating random cuts of a sphere with chosen diameter. Finally, to make a comparison, the pore volumes were calculated employing stereological equations.

Key-Words: - Foams, Plane cut visualization, Voronoi tessellation, Image analysis, Modelling the structure, Pore size distribution

1 Introduction

Studying and determining the characteristics of cellular materials has become a popular task in the field of stereology. Many recent studies have focused on determination of pore size distribution (PDS) of different types of porous materials, such as foams or various grainy structures, using multiple methods [1-3]. The reason of this trend is undoubtedly the raising application range of these materials – particularly in civil engineering and packaging industry. These materials are so popular because of their low specific weight in combination with reasonable mechanical properties.

Employing either the mathematical and computational tools [4] or the methods for direct observation of the structure [3], the investigators study the pore size distribution as a parameter determining the particular property of given material for certain application. Thus, the research in this area is restricted rather to materials engineering.

However, according the definition of foam, also some food products – such as pastry, fall to this category. This study deals with the method, which has been developed for the evaluation of pore size distribution in soft polyurethane foams [2] and proposes its usage in food industry as a cheap quality control tool.

2 Theory

Two basic stereological tools for image analysis were employed:

2.1 Euclidean distance map

Euclidean distance map (EDM) is widely used tool for image analysis [5]. EDM is computed from strictly binary images of the analyzed structure, where the foreground is usually represented by black pixels, while the background is white.

The obtained images of the structure White (empty) pixels of background are characterized by the value $b_{pq} = 0$, black pixels of foreground objects have value $b_{pq} = 1$. Based on these values, EDM computes the distance of each individual white pixel to the closest black pixel as follows:

$$d_{ij} = \min_{1 \le p,q \le N} \left(\sqrt{(p-i)^2 + (q-j)^2} \mid b_{pq} = 1 \right) \quad (1)$$

EDM is presented in the graphical form, where the background vacancies between the foreground objects are illustrated by tint of gray based on the distance from the foreground (Fig. 3). The bigger distance, is indicated by the lighter tint of gray. The lightest areas located in the centers of the vacancies are EDM maxima and can be interpreted as the pore centers.

2.2 Voronoi tessellation

Voronoi tessellation (VT) is a way of decomposition of any area to finite number of cells with mutual boundaries, which is based on the same number of tessellation generators. VT is also called Voronoi decomposition or Dirichlet tessellation.

Let $P = \{p_1, p_2, \ldots, p_n\}$ is a set of points in a d-dimensional space $\mathbf{R}^d$ (in the case of plane $d = 2$). The Voronoi tessellation for P is a division of the space, which associates a region $V(p_i)$ with each tessellation generator p_i from P in such a way, that all points in $V(p_i)$ are closer to p_i than to any other point in the P. This can be described by following mathematical formula:

$$V(p_i) = \left\{ x \in \mathbf{R}^d, \left\| x - p_i \right\| \le \left\| x - p_j \right\| \right\} \quad (2)$$

where $x \ne j$ and $\|x - p_i\|$ is Euclidean distance. The unification of all cells of $V(p_i)$ is a general Voronoi tessellation [6].

Areas of cells based on Voronoi tessellation can be easily computed and used as an approximation of areas of planar section of pores.

2.3 Pore volumes approximation

Values of cell areas obtained by VT are only *planar* characteristic and do not provide the complete information about the structure of the material. Thus, in order to describe the structure *spatially*, pore volumes must be calculated. There are several methods for calculating and/or approximating the volume of pores; all of them suppose semi-spherical shape of pores [5].

According to the stereological f_D-function estimation method, which is based on Wicksell's equation (3) [7], a pore can be modeled as a sphere of diameter D. The sphere diameter distribution can be characterized by probability density function (PDF) f_D and the mean diameter $\mathbf{E}D$. Planar section of the sphere is a circle profile of diameter d. The profile diameter distribution is in analogy characterized by the PDF f_d, the mean value $\mathbf{E}d$.

$$f_d(d) = \frac{d}{\mathbf{E}d} \int_d^{D_m} \frac{f_D(D)}{\sqrt{D^2 - d^2}} \mathrm{d}D \quad (3)$$

The equation is valid for $0 \le d \le D_m$, where D_m is the maximum of D and d.

Another method, which is standardized [8] suggests the equation for calculation of the mean pore volume $\mathbf{E}V$ from the mean pore section area $\mathbf{E}a$:

$$\mathbf{E}V = C'.(\mathbf{E}a)^{3/2} \quad (4)$$

Where:

$$C' = \sqrt{\frac{6}{\pi}} = 1.382 \quad (5)$$

3 Experimental

Eleven samples of bread with varying ratio wheat/rye flour content were tested in order to estimate the mean pore volume and distribution of pore sizes.

3.1 Baking the samples

The bread samples were prepared according to ICC standard no. 131 [9]. The dough was made in a specified mixer from flour, water, dry yeast, salt, sucrose and ascorbic acid. Dough pieces were scaled, rounded, rested for 30 min, consequently sheeted and moulded, afterwards placed in tins, proofed for 50 min and finally baked for 20 min.

3.2 Test pieces

The samples were cut on 1.5-2 cm thick slices and dried in ambient conditions of the laboratory during three days. Consequently the surface of each test piece was grinded by fine sandpaper to ensure totally plane surface for further image analysis.

The test pieces were covered by very thin layer of stamp ink on the surface and scanned, employing a common scanner device. The scanning resolution was set on 1000 dpi.

3.3 Density of samples

Prior to the image analysis, density of samples was determined. The base area of the test pieces in pixels was assessed in picture-editing software (GIMP). The thickness was measured on the test pieces using digital micrometer. All samples were weighed on laboratory balances and finally the density was calculated from the knowledge of weight and volume of the test pieces. The density of all measured samples is shown in Table 1:

Table 1: Density of the samples (dried).

Sample		density [g.cm^{-3}]
nr.	wheat/rye [%]	
T100	100/0	0.38
TS9010	90/10	0.35
TS8020	80/20	0.41
TS7030	70/30	0.50
TS6040	60/40	0.52
TS5050	50/50	0.53
TS4060	40/60	0.56
TS3070	30/70	0.61
TS2080	20/80	0.72
TS1090	10/90	0.71
S100	0/100	0.79

According to Table 1 one can conclude, that the density rises with raising content of rye flour. Compared to the pure-wheat dough, the pure-rye dough has approximately two times higher density.

3.4 Image analysis

Image analysis was carried out on the images obtained by scanning. The original scans (Fig. 1) were edited in GIMP – color images were transferred into black and white ones, and the binary threshold was set to the values avoiding the deep holes (Fig. 2).

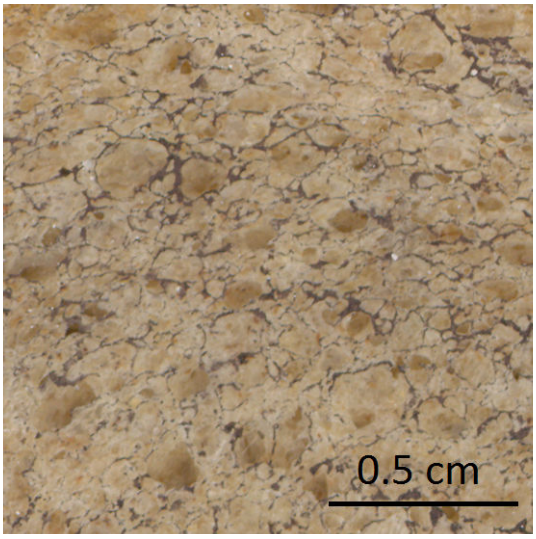

Fig. 1: Surface scan of the bread, sample TS5050.

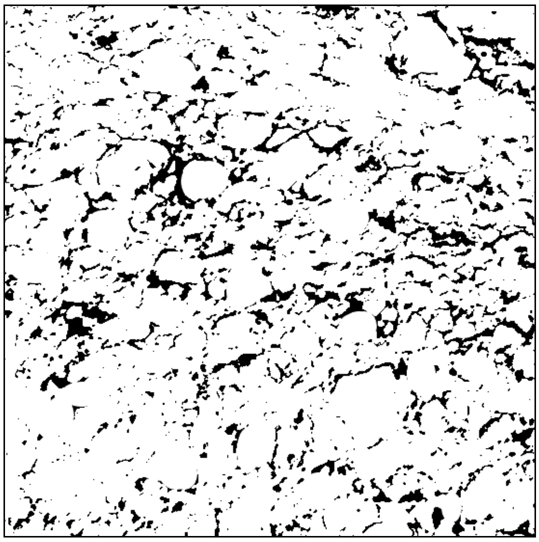

Fig. 2: Threshold image of the scan, sample TS5050.

These images were consequently evaluated employing EDM (Fig. 3). That was applied on the image with inverted colors. Local maxima of EDM, which can be understood as pore sections centers, were approximated by circles with the centers located in the maxima (Fig. 4). This approximation does not fit the vacancies well. For this reason the second approximation based on VT was carried out (Fig. 5). VT generated from the centers of the circles provides a real-like net, approximating the structure better than circles. Moreover the areas of pore section and their distribution (Fig. 6) are known.

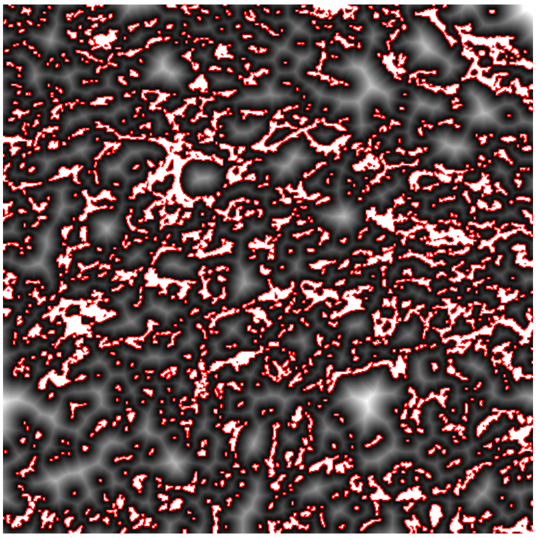

Fig. 3: Euclidean distance map (inverted colors), sample TS5050.

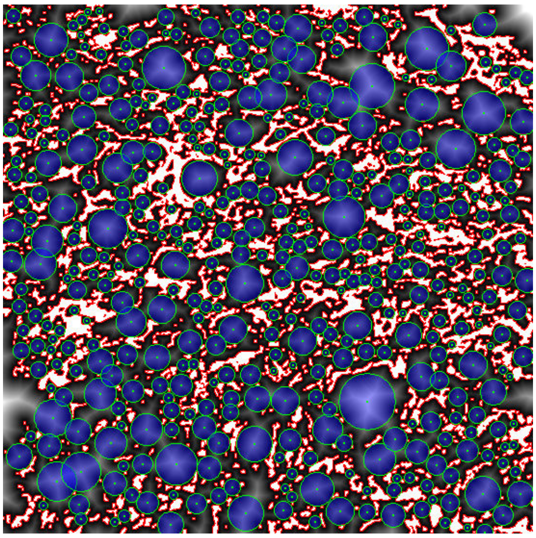

Fig. 4: First approximation of the EDM by circles, sample TS5050.

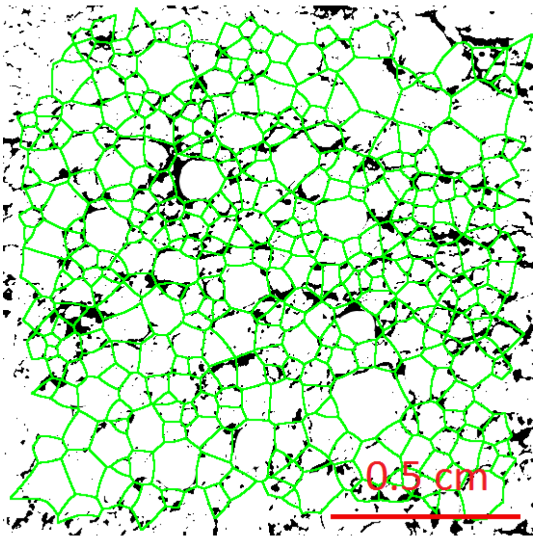

Fig. 5: Second approximation of the EDM by VT, sample TS5050.

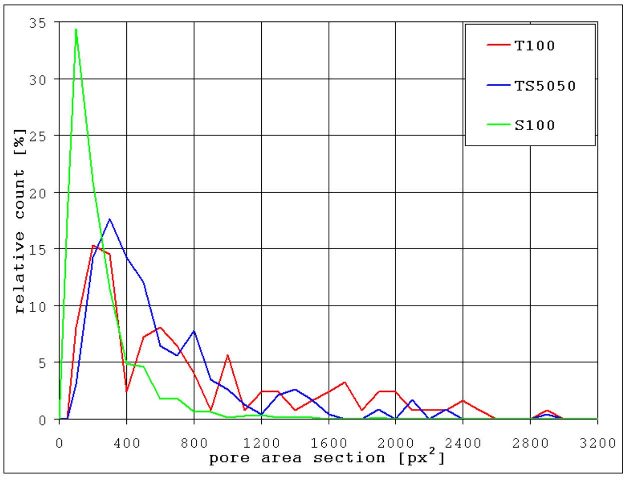

Fig. 6: Pore section distribution of the samples T100, TS5050, S100

Fig. 6 shows a comparison of pore section area distributions of three selected samples – T100, TS5050 and S100. As can be seen, the raising content of rye flour “pushes” the distribution curve more to the left. Moreover it modifies the width of the distribution.

3.5 Model

VT approximates only the planar section of porous structure. In order to know the distribution of pore volumes a computer simulation was carried out. The principle of the simulation was generating large number of spheres with normal random distribution of radii $N(\mathbf{E}r, \sigma^2)$. These spheres were cut by random planes and distribution of model planar section areas was obtained. Employing Monte-Carlo method the optimal $\mathbf{E}r$ and σ^2, best approximating experimental distribution of planar section of areas, were looked for (Fig 7).

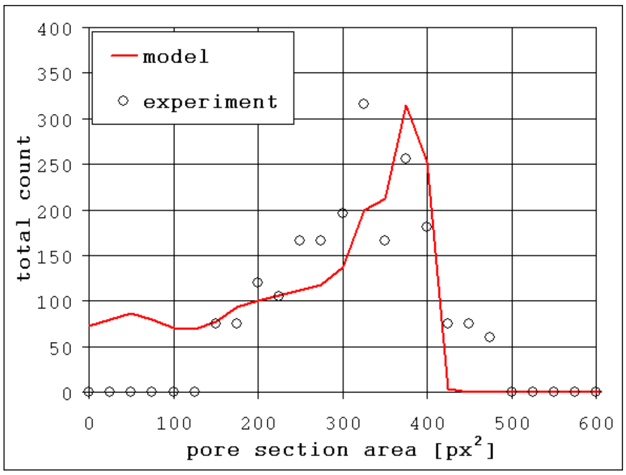

Fig. 7: Experimental data fitted by the model, sample TS5050.

It is obvious from Fig. 7, that the model does not fit the experimental values well in the region approx. 0-150 (x-axis). This disagreement is caused due to the fact that the small pore sections can be hardly visualized and therefore do not appear in the graph. It is also clear, that the distribution is quite narrow.

3.6 Pore volumes

In order to compare different approaches of computing the mean volume of pores, three methods of calculation of the mean pore volumes were employed.

The principle of the first one ($\mathbf{E}V_1$) was calculating the mean volume of the sphere with the average radius obtained from the model.

The second approach ($\mathbf{E}V_2$) was based on the calculating the mean volume by making the average from the volumes of the model spheres.

Finally, the third method of calculation of the mean volume ($\mathbf{E}V_3$) employed the stereological equations (4) and (5) and transferred the mean pore sections areas into the mean volume of pores. The results for the three selected samples T100, TS5050 and S100 are summarized below, in Table 2:

Table 2: Mean volumes of the samples T100, TS5050, S100

Sample	**model**		$\mathbf{E}V_1$ [mm^3]	$\mathbf{E}V_2$ [mm^3]	$\mathbf{E}V_3$ [mm^3]
	$\mathbf{E}r$ [mm]	σ			
T100	0.56	0.686	0.75	4.14	0.66
TS5050	0.29	0.003	0.10	0.10	0.39
S100	0.14	0.003	0.01	0.01	0.09

As the methods for calculation of $\mathbf{E}V$ are different, the results shown in Table 2 are different too. This leads to the conclusion that the information about mean pore volume of any material must be always accompanied with the description how the value was obtained.

The value $\mathbf{E}V_2$ of the sample T100 is much higher than the others. It is affected by wide distribution of pore radiuses r (high value σ in the 3rd column of Table 2) which leads to occurence of very big pores moving mean value to high numbers.

4 Conclusion

The usability of the method for estimation of pore sizes in polymer foams with opened cells has been successfully verified also for the usage on samples of pastry.

According to the presented results, two statements can be concluded.

Table 1 shows, that the raising content of rye flour brings higher density of the bread. It is not surprising that the higher density corresponds with smaller pores, which is confirmed in Table 2.

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