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PARTICLE SIZE DISTRIBUTION OF RE-USED CARBONATION MUD

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Key words: carbonation mud, particle size distribution, hydrocyclones, wastes from food technology, image analysis

Carbonation mud contains $CaCO_3$ and aggregated or adsorbed substances from sugar beet raw juice. The precipitate can be characterised as a polydisperse system. Large volumes of carbonation mud predetermine its use as a fertilizer. Other possibilities of utilization are tested – *e.g.* addition in animal feed mixtures, application as paper, plastic and rubber fillers, and usage for the building industry and for desulphurization of combustion gases. Re-using of carbonation mud in sugar technology enables to decrease lime consumption in a sugar factory and to minimize carbonation mud production. It involves economic aspects and environmental effects (decreasing lime stone mining and limiting carbonation mud production).

For these feasible internal or external applications of carbonation mud it is necessary to know size distribution in advance. The research work was aimed at particle size distribution measurement of carbonation mud suspension, which was separated in hydrocyclones used in MZ-technology. This low-cost sugar juice purification method is based on re-using carbonation mud and following separation of mud particles. Authors have chosen an image analysis method (system LUCIA) combined with microscopic observation for particle equivalent diameter determination.

At first it was necessary to work out original measuring methodology for carbonation mud, it includes choosing suitable object-lenses, design of lightning, preparing suitable subroutine in which values of contrast and threshold are defined, *etc.* From these size data the particle size distribution was calculated.

We detected that the industrial method for the preparation of input mud suspension was not fine enough and an amount of small particles increased in comparison with the unfiltered 1st carbonation juice. Bottom output suspension of hydrocyclones was of worse quality with small particles than the 1st carbonation juice as well. Hydrocyclones did not affect small particles.

A normality of size distribution of particles from input and output suspensions in MZ-technology was tested. It can be characterised by normal distribution function for volume fraction x_V or by log-normal distribution function for frequency of particles, as to input particles and upper output particles of hydrocyclones. The measured results will be groundwork for next carbonation mud applications.

SYMBOLS AND UNITS

A (μm^2) – scanned cross-area of the particle; d_e (μm) – equivalent diameter of the particle; DS (%) – dry substance; n (1) – frequency, number of particles; np (1) – theoretical (expected) frequency, number of particles; pCaO (g CaO/100 mL) – lime addition; Sd (g/kg) – solids content; x_V (%) – volume fraction of the interval in relation to whole volume of solids; δ (–) – standard deviation; χ^2 (–) – statistical characteristic defined by equation (2)

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j – interval sequence; k – number of intervals

INTRODUCTION

An addition of lime milk into sugar beet raw juice coagulates colloid substances and precipitates non-soluble or hardly soluble substances. Subsequently the lime surplus is precipitated by carbon dioxide and originating 1st carbonation juice or carbonation slurry concentrate is filtered (Figure 1). The precipitate, called carbonation mud, contains fine crystals of $CaCO_3$ and aggregated or adsorbed non-sugars; *e.g.* phosphates, magnesium, potassium and calcium salts, trace elements, nitrogenous and other organic compounds, including colour substances.

Industrial and agricultural application of carbonation mud

Great volumes of carbonation mud predetermine its use as a product – fertilizer. Tank cars transport liquid carbonation mud to fields for fertilization, where it is pumped to sprayers. It is necessary to dose about 10 t/ha of carbonation mud (DS = 70%) to increase soil pH from 6.0 to 6.5 or 7.0. Carbonation mud helps to increase calcium content; it fastens stabile mould structure and thereby supports active life in soil.

There was tested an addition of carbonation mud to feed mixtures where it supplies calcium for nutrition of animals. But it is necessary to have veterinary and legal approval for this application.

Carbonation mud can be used for cement and breeze-blocks production, *etc*. However, some residual sugar content in mud can inhibit solidification of concrete or mortar mixtures.

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Other application possibility is as filler – substituting milled or powdered $CaCO_3$. This calcium carbonate application possibility for plastics (polyethylene, polypropylene *etc*.), rubber and paper is well known but it is the research object henceforth [Rungruang *et al.*, 2006; Teixeira *et al.*, 2005; Cai *et al.*, 2003; Xu & Pelton, 2005; Kotek *et al.*, 2004]. The trend of applying $CaCO_3$ as a filler in graphic papers is increasing. The size distribution criteria are very important for all these applications.

Fillers for plastics and rubbers could be produced from carbonizated carbonation mud, which originates by heating up to 600–700°C. It is a grey powder of maximum size of 60 μm consisting of agglomerated carbon and microcrystallic $CaCO_3$. Agglomerated particles are bigger in comparison with precipitated particles of $CaCO_3$ with the size of 0.2–0.5 μm. But the vulcanisation and application properties in rubber mixtures or in vulcanised material are not worse. Physico-chemical properties of mud used into polypropylene are suitable if the size particle is in a range of 2–3 μm [Smelik & Fuzy, 1988]. Also it is necessary to process it not to contain unsuitable substances [Smelik *et al.*, 1990].

An environmental application is the use of mud for desulphurization of combustible gases, which is strictly required in some countries. The use of carbonation mud for fuel oil burners has been investigated and put in technical operation by Dolignier & Martin [1997].

Re-using of carbonation mud to decrease lime consumption in a sugar factory

Compressibility coefficient and specific resistance of the filter cake are decisive factors of filterability of sugar juices. It is usually applied that specific resistance of a filter cake is proportional to reciprocal square of particle diameter for the monodisperse system. High resistance of cake due small particles implicates decreasing filter performance. Recently Grabka *et al.* [2002] studied the measurement of compressibility coefficient.

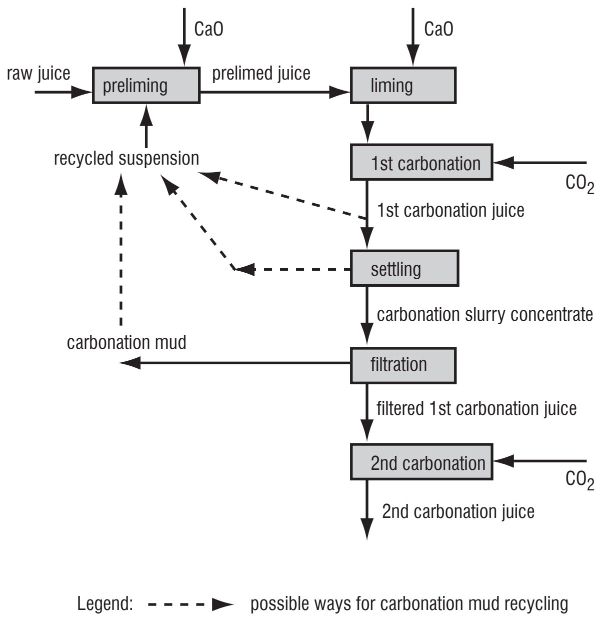

FIGURE 1. A simplified scheme of the first part of juice purification.

A settler is used before filtration (see technological schema in Figure 1). Classical Stokes' law is valid where settling rate is proportional to the square of particle diameter.

The size of particles, their filterability and settling ability, can be influenced by lime addition above all. Very low lime addition may result beyond the difficult filterability in worse juice colour and hardness [Buchholz & Bruhns, 1996]. It is necessary to prevent particle damage in following technology – *e.g.* in pumps (wrong design or technical state). Other errors can be unsuitable quality of raw juice or wrong alkalinity of the 1st carbonation juice (Figure 1).

Realization of such sugar technology, which creates suspension with sizeable particles of carbonation mud, enables to decrease lime consumption in a sugar factory and to minimize carbonation mud production. It involves environmental effects – limiting of smelling carbonation mud production, storage and disposal, decreasing limestone and pit-coal mining, decreasing of emission of superfluous CO_2. Except these ecological effects there are economical outputs for a sugar factory – decreasing operating costs (direct inputs, transport costs) and reduction of investment costs for factory extension (it is not necessary to build new lime-kiln).

These effects can be achieved by different economic methods of juice purification using recycling carbonation mud for preliming (see Figure 1). These methods provide a sufficient size of carbonation mud particles.

The simplest economical method of juice purification is the recycling of the 1st carbonation juice [Kovařík & Rohlík, 1973]. A good filterability of the 1st carbonation juice should be secured by the ratio pCaO/Sd lower than 0.03, where pCaO is lime addition (g CaO/100 mL) and Sd (g/kg) is solids content [Kadlec *et al*., 1983].

For heat energy savings it is more suitable to recycle carbonation mud suspension and not the 1st carbonation juice. Many low-cost methods of juice purification including MZ method are based on this principle [Sarka, 1999; Grabka & Baryga, 2002; Moc & Záruba 1993, 1995].

MZ-method used in the sugar factory Vrdy

Our study is focused on the method MZ installed in the sugar factory Vrdy, Czech Republic. This sugar raw juice purification technology is based on re-using carbonation mud and following separation of mud particles. The simplified schema is shown in Figure 2.

Description of the scheme:

Carbonation slurry concentrate is filtered by filter-presses (1) and washed carbonation mud is mixed with water in a mixer (3). The density is controlled and the pollution and lumps are separated by vibratory sieve (4). The resulted suspension C is concentrated and separated by hydrocyclones (6, see Figure 3), more dense suspension with bigger particles (E) from the bottom of cyclones is dosed together with lime milk to the reactor (7). Suspension from the reactor is dosed to the last chamber of prelimer (see Figure 1). Waste water with small particles (suspension D) goes to flume water where it increases pH and therefore prevents from microbial infection.

The advantage of this method in comparison with other methods is that the carbonation mud is mixed with water without any sugar juice. Thus the products are transferable for other applications of carbonation mud.

Hydrocyclones are currently used in food industry, *e.g.*

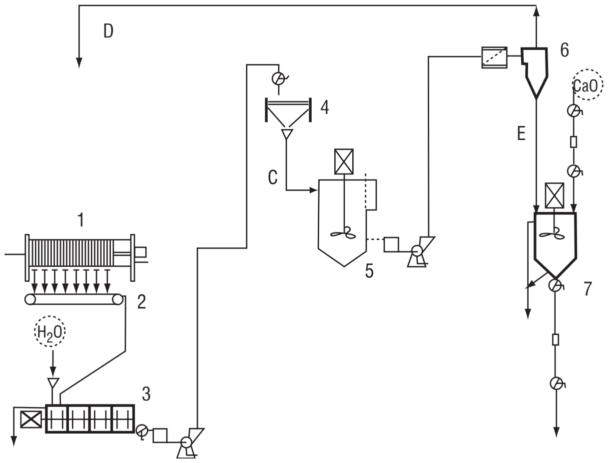

FIGURE 2. Simplified scheme of MZ-method at sugar factory Vrdy. Legend: 1 – filter press, 2 – mud conveyer, 3,5 – mixers, 4 – sieve, 6 – hydrocyclones, 7 – reactor; C, D, E – points of suspensions sampling

FIGURE 3. The battery of hydrocyclones in MZ-technology.

when slaking lime in sugar industry or starch fractionation. Many authors took interest in theoretical description of hydrocyclones, *e.g.* Kutepov *et al.* [1987], Trawinski [2003] and Ortega-Rivas [2006]. The Tasco juice purification system [Mc Ginnis, 1971] used hydrocyclones to produce carbonation slurry which was recycled to the preliming. Athenstedt [1965] described factory trials with hydrocyclones.

The research work was aimed at size distribution measurement of carbonation mud suspensions separated by hydrocyclones. The objects of observations were: (i) to prepare methodology for carbonation mud particle size measurement by image analysis (instrument LUCIA), (ii) to evaluate quality of suspension C (see Figure 2) entering hydrocyclones and output suspension E (see Figure 2) returned to technological process, and (iii) to check if there are some damages of particles by hydrocyclone operation.

Fulfilments of these objects were important to get a standard quality of the product.

METHODS

There are many methods for particle parameter determination – optical or electron microscopy, sedimentation, specific area measuring *etc*. In the Czech Republic the microscopic method for carbonation mud was used by Osvald [1959], the size of particles was about 2 μm. Sarka [1999] presented microphotographs of carbonation mud from a scanning electron microscope with magnification of 30000.

Nowadays, an image analysis is used for many applications. The majority of the applications of computer vision in the food industry focus on food quality and grading [Sun, 2006], evaluation of mixing [Tukiendorf *et al.*, 2003] and separation processes and evaluation of food texture and microstructure [Sadowska *et al.*, 1999; Svec & Hruskova 2004]. Image analysis can be used for particle size determination as well. Bubnik *et al.* [2000, 2001] measured parameters of sugar crystals using the instrument LUCIA.

For evaluation of microscopic observation of particles we used the imaging system LUCIA G/Comet V3.52 (=Laboratory Universal Computer Image Analysis; product of the LABORATORY IMAGING Co. Prague, Czech Republic) with a personal computer, processor Pentium® (150 MHz, 32 MB RAM). A camera Cohu 2252 TV CCD placed on an adjustable stand KAISER RS1 is able to transfer data to the computer. The main advantage of the system is a possibility to evaluate a vast number of particles by the computer. All performed steps were automatically saved in a subroutine and the whole process thus could be repeated if needed. Some steps can be changed or deleted. When the control of automatic evaluation is necessary, program can be stopped and then modified or confirmed.

Before measurement, it is necessary to choose optimal hardware components: lighting setting: transparent or non-transparent objects, lighting from different angles (flexible optical fibre), and combination of camera with object-lenses. For the measurement of carbonation mud we used controllable central and two point source lights and black background. We operated with two object-lenses with magnification of 4.5 and 2 in series.

The basic principles of measurement are presented in Figure 4. After TV image is captured it is converted into a grid of tiny "pixels", which we can imagine as electronic equivalents of stitches in a tapestry. The total proportion of the image that is dark can be recorded by counting the number of light and dark pixels. It has the potential advantage if the size of each particle is measured. Measured and evaluated objects are placed in the so-called "measuring frame". When the whole object is measured, the whole image is evaluated; however particles touching the frame are excluded from evaluation.

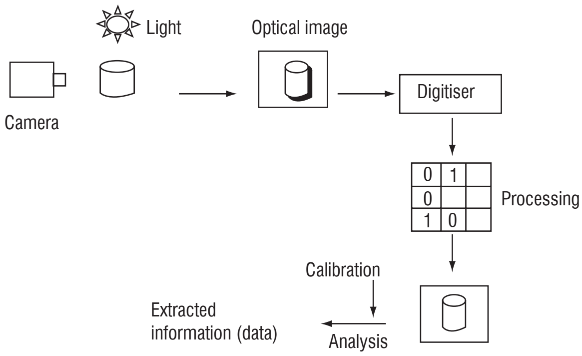

FIGURE 4. Principles of image analysis for particle size measurement, modified scheme according to Sun [2006].

Before evaluation it is necessary to adjust contrast of actual colour image and to define parameters for segmentation of the colour image according to threshold values (see Figure 5). These parameters were read automatically during the subroutine course, but it was necessary to correct the values for every suspension. It was necessary to ensure an optimal number of particles in a measuring frame as well. For this purpose, we diluted the 1st carbonation juice with filtered one at a 1:1 ratio. For denser suspensions the dilution ratio is proportional to their concentration.

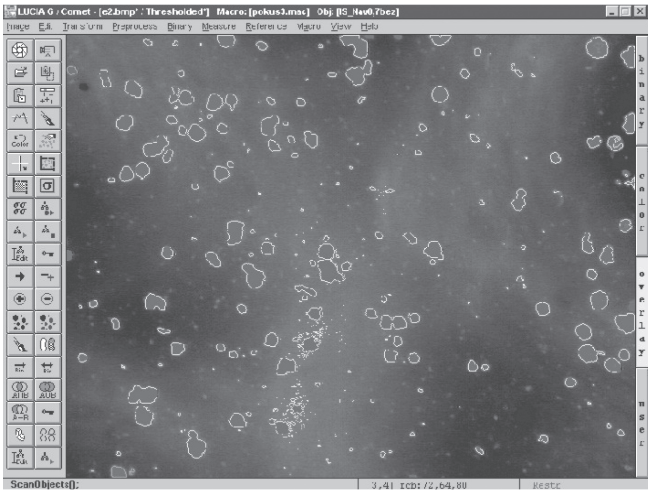

FIGURE 5. Scanned objects of the suspension after contrast and colour interface adjustment.

To have extracted information it is necessary to make a calibration of instrumental readings. For every chosen combination of object-lenses it is necessary to do individual calibration which can be saved in a computer and used again. A precise calibration is highly important for the measurement. It gives the real size of measured objects. A special calibrating glass grid LUCIA with different scales was used for calibration. Every scanned object area was measured and equivalent diameter d_e was calculated after calibration:

$$d_e \equiv \sqrt{\frac{4A}{\pi}} \qquad (1),$$

where: A is an area of measured object (μm^2). The equivalent diameter d_e specifies the characteristic size of particles of carbonation mud.

Minimal size of the measured object was about 1 μm, but we evaluated objects with d_e bigger than 2 μm to avoid background noise. It is evident from the results that this resolution was fully true.

The ability to separate and differentiate the object is a basic process for an object measurement. It is necessary to check all objects and to exclude not-suitable objects (bubbles, fibres *etc.*) from the evaluation. Control points during the subroutine make it possible.

All results were processed and statistically evaluated. The program LUCIA calculated basic characteristics – mean value, standard deviation, minimal and maximal value. The output of the measurement was a histogram (in numerical and graphical forms) as well - particle sizes (equivalent diameters) were divided in intervals and their numbers in every interval (*i.e.* frequency) was calculated.

We measured particle dimensions in one sample of suspension in about 10–20 measuring frames. The total number of measured objects (n) for every suspension was in between 1000-3200. Data in these histograms were used for next statistic evaluation, because simple statistics was not sufficient. We used the obtained histograms to create a wide histogram for every suspension and calculate the total size particle distribution.

Except this type of histogram we also created histograms where instead of the frequency we evaluated the volume fraction for every size interval. This type of histogram is analogous if we sieve the particles and weigh the separate sieve fractions, of course, when solids are homogenous. The volume fraction is equal to the mass fraction then.

The measurement of particles size measurement was completed by analysis of solid concentration and by mass balance of the process shown in Figure 2.

We tried to compare the obtained distribution with two distribution models: A. Normal distribution function for volume fraction x_V; and B. Log-normal distribution function for particle frequency.

This comparison was realized by calculating of χ^2, where the statistic probable values of frequency (or volume fraction) are computed according to the equation 2:

$$\chi^2 = \sum_{j}^{k} \frac{(n_j - np_j)^2}{np_j} \qquad (2),$$

where: n_j – frequency (in case A volume fraction), np_j – theoretical (expected) frequency (in case A volume fraction). The value χ^2 was compared with criterion χ^2 in tables.

For the distribution function A, where we operate with volume fraction, there does not exist the needed number n for calculation of standard deviation (δ). At first we estimated δ for n=100 and then we evaluated it by non-linear regression. A distribution B is used for very small particle size with wide variance; an example is a similar distribution for powder sugar [Gebler, 1983].

RESULTS AND DISCUSSION

The first target was a comparison between distribution of the 1st carbonation juice (see Figure 1) and suspension C (see

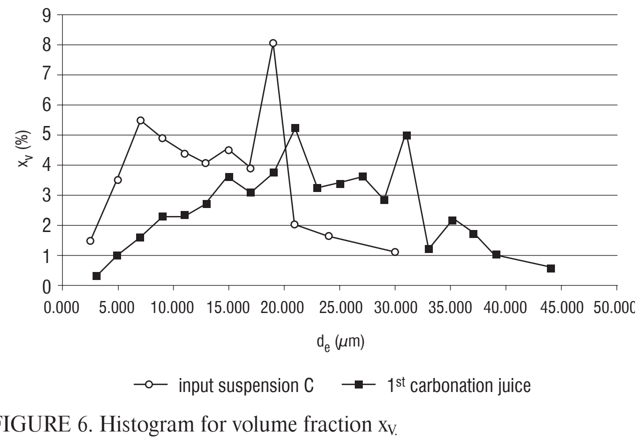

FIGURE 6. Histogram for volume fraction x_V.

Figure 2). The resulted histogram (Figure 6) shows that the primary manipulation with mud in MZ-technology was very abrasive with many resulting small particles.

The following histogram (Figure 7) illustrates results of suspensions C, D, and E (according to Figure 2).

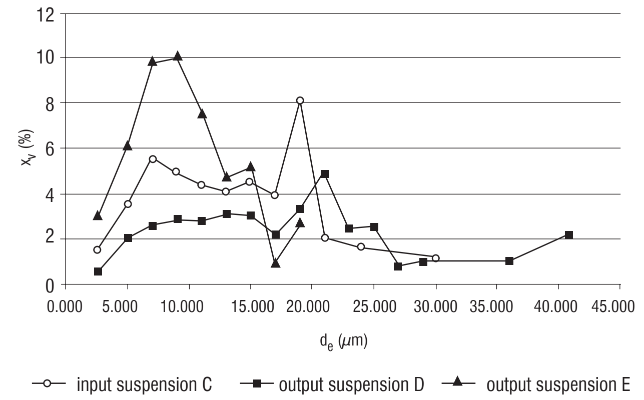

FIGURE 7. Histogram for volume fraction x_V.

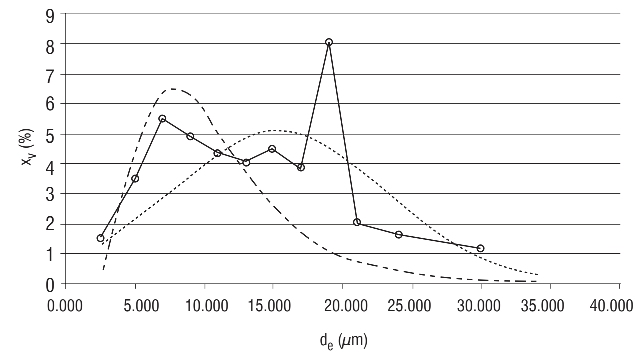

FIGURE 8. A comparison of histogram of suspension C with the model of normal distribution A (--) and log-normal distribution B (–‥–).

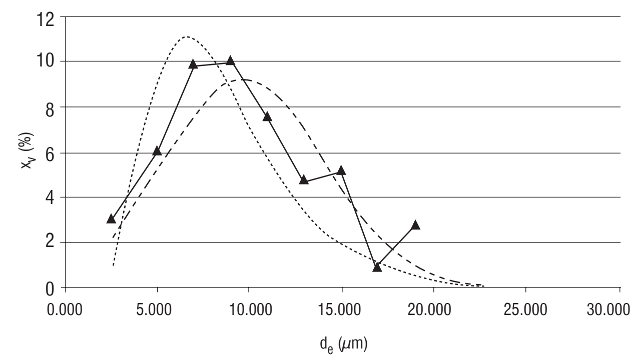

FIGURE 9. A comparison of histogram of suspension D with the model of normal distribution A (--) and log-normal distribution B (–‥–).

Suspension D output from hydrocyclones contained a significant fraction of small particles conformable with expected behaviour of a hydrocyclone. Bigger particles prevail in suspension E. The measurement was completed by mass balance of the chosen technological node for every size interval. Compared with Madsen *et al.* [1998], we did not discover any significant increase or decrease of any fraction.

Both hypotheses of A and B distributions are suitable (Figures 8 and 9, Table 1) for the suspensions C and D (see scheme in the Figure 2). Normal distribution A is more fitting in both cases (like sum of squared residuals). Any of this distribution did not agree with data of suspension E (the real distribution was too flat).

From the technological point of view it is important that suspension E be at least of the same size of particles as the1st carbonation juice (see the schema in the Figure 1) to be returned as a carbonation slurry back to sugar technology. Measured results (see Figure 10) show that output suspension E did not contain bigger particles due to the hydrocyclone separation and did not fulfil quality requirements exactly.

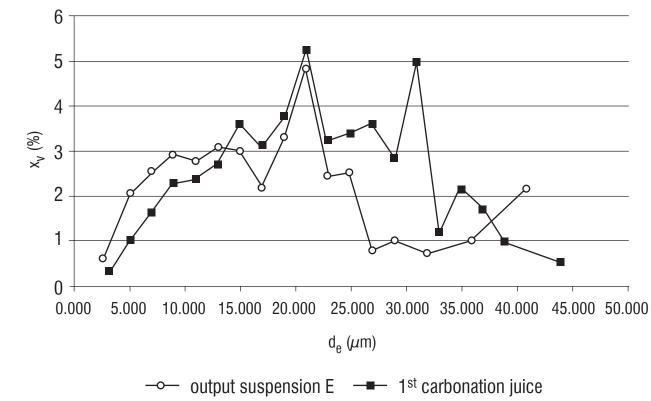

FIGURE 10. A histogram for volume fraction x_V.

CONCLUSIONS

The research work was aimed at particle size distribution testing for suspensions of carbonation mud in the system MZ in which the mud was mixed with water and it was separated by hydrocyclones. For particle size measuring use was made of the image analysis method combined with microscopic observation by the system LUCIA. The measured results will be groundwork for next carbonation mud applications.

At first it was necessary to work out original measuring methodology for carbonation mud, it includes choosing suitable object-lenses, design of lightning, preparing suit-

TABLE 1. Statistic parameters for suspensions and distribution A and B, significance $\alpha = 0.05$.

	Distribution A				Distribution B			
Suspension	Mean d_e	Standard deviation d_e (σ)	χ^2	Criterion χ^2	Mean ln d_e	Standard deviation ln d_e (σ)	χ^2	Criterion χ^2
C	15.3	7.9	14.82	15.50	1.570	0.485	6.72	11.07
D	9.7	4.3	5.80	16.91	1.506	0.438	7.86	11.07
E	25.4	17.0	22.84	12.59	1.644	0.525	6.00	5.99

able subroutine in which values of contrast and threshold are defined, *etc*.

Measured results are as follows:

1. The industrial method for the preparation of mud suspension (C) was not fine enough and increased an amount of small particles in comparison with the unfiltered 1st carbonation juice.

2. Bottom output of hydrocyclones (E) was of worse quality than the 1st carbonation juice. It contained more small particles.

3. Hydrocyclones did not crush down small particles to small ones.

4. A normality of size distribution of particles from input and output suspensions in MZ-technology was tested. It can be characterised by normal distribution function for volume fraction x_V or by log-normal distribution function for frequency of particles, as to the big particle distribution of (C) and upper output of hydrocyclones (D).

A further research and measurement to obtain a wide range of various suspensions of carbonation mud will be necessary.

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REFERENCES

1. Athenstedt M., Über die Verwendung von Hydrozyklonen bei der Saftreinigung. Z. Zuckerind., 1965, 15, 456–458 (in German).
2. Bubnik Z., Kadlec P., Pour V., Hinkova A., Image Analysis of sugar crystals in massecuite and dry state – Computer Image Analysis: a powerful tool for evaluation and control of the crystallization process. 4th Symposium of ANDREW VAN HOOK ASSOCIATION. 29 March 2001, Reims, France. Extended abstract: Zuckerind. 2001, 126, p. 215.
3. Bubnik Z., Kadlec P., Starhova H., Pour V., Hinkova A., Application of computer image analysis for evaluation and control of the sucrose crystallization process. Czech J. Food Sci., 2000, 18, 23–28.
4. Buchholz K., Bruhns M., Über die Kampagne 1995/96 und neuere technologische Entwicklungen. Zuckerindustrie, 1996, 121, 305–326 (in German).
5. Cai H.H., Li S.D., Tian G.R. Wang H.B., Wang J.H., Reinforcement of natural rubber latex film by ultrafine calcium carbonate. J. Appl. Polym. Sci., 2003, 87, 982–985.
6. Dolignier J.C., Martin G., High-temperature flue-gas desulfurization by injection of carbonatation lime. Zuckerind., 1997, 122, 927–933.
7. Gebler J., Výběr geometrické řady sít. Listy cukrov., 1983, 99, 273-275 (in Czech).
8. Grabka J., Baryga A., Možnosti zvýšení kvality šťávy při epuraci s aktivovaným kalem. Listy cukrov. a řep., 2002, 118, 218–223 (in Czech, English abstract).
9. Grabka J., Brzeziński S., Pałka A., Stanovení součinitele stačitelnosti sraženiny při průmyslové výrobě cukru. Listy cukrov. a řep., 2002, 118, 259–261 (in Czech, English abstract).
10. Kadlec P., Bubnik Z., Svoboda A., Sarka E., Měření I. saturace v cukrovaru Hrochův Týnec. Listy cukrov., 1983, 99, 199–201 (in Czech).
11. Kotek J., Kelnar I., Baldrian J., Raab M., Tensile behaviour of isotactic polypropylene modified by specific nucleation and active fillers. Eur. Polym. J., 2004, 40, 679–684.
12. Kovařík A., Rohlík M., Ověření některých variant čištění difúzní šťávy. Listy cukrov., 1973, 89, 33–41 (in Czech).
13. Kutepov A.M., Ternovsky I.G., Tsyganov L.G., The calculating method of flow characteristics and separating efficiency of turbocyclones. Congress CHISA, 1987, Prague, Czech Republic.
14. Madsen R.F. chapter 9.3.3., 1998, *in*: Sugar Technology. Beet and Cane Sugar Manufacture (eds. P.W. van der Poel, H. Schiweck, T. Schwartz). Verlag Dr. Albert Bartens KG, Berlin, pp. 529–544.
15. Mc Ginnis R.A., Beet Sugar Technology. 2. ed. 1971, Beet Sugar Development Foundation, Fort Collins, pp. 191–232.
16. Moc D., Záruba J., Regenerace saturačního kalu. Listy cukrov. a řep., 1993, 109, 41 (in Czech).
17. Moc D., Záruba J., The MZ method of raw juice purification. Int. Sugar J., 1995, 97, 287–291.
18. Ortega-Rivas E., Theoretical model of hydrocyclone separation for application in food suspensions. 2006; *in*: Proceedings of the 2nd CIGR Section VI International Symposium on FUTURE OF FOOD ENGINEERING, 26-28 April 2006, Warsaw, Poland.
19. Osvald R., Mikroskopické hodnocení kalových částic získaných na první saturaci při různých epuračních metodách. Listy cukrov., 1959, 75, 275 (in Czech).
20. Rungruang P., Grady B.P., Supaphol P., Surface-modified calcium carbonate particles by admicellar polymerization to be used as filler for isotactic polypropylene. Colloid Surface, 2006, A 275, 114–125
21. Sadowska J., Jeliński, T., Fornal J., Comparison of microstructure of vitreous and mealy kernels of hard and soft wheat. Pol. J. Food Nutr. Sci., 1999, 8/49, No. 4, 3–15.
22. Sarka E., Activation of carbonation slurry concentrate – a method of saving limestone. Zuckerindustrie, 1999, 124, 411–414.
23. Smelik A., Fuzy S., Spôsob čistenia tuhých práškových anorganických plniv. PV ČSSR No. 4994-88, 1988 (in Slovak).
24. Smelik A., Halasova G., Fuzy S., Úprava karbonizovaného saturačního kalu na aplikáciu v polymérnych kompozitech. Listy cukrov., 1990, 106, 110–114 (in Slovak).
25. Sun D.W., Application of computer vision for food quality evaluation. 2006, *in*: Proceedings of the 2nd CIGR Section VI International Symposium on FUTURE OF FOOD ENGINEERING, 26–28 April 2006, Warsaw, Poland.
26. Svec I., Hruskova M., Image data of bread crumb structure from Czech spring wheat cultivars. Czech J. Food Sci., 2004, 22, 133–142.
27. Teixeira S.C.S., Moreira M.M., Lima A.P. Santos L.S., da Rocha B. M., de Lima E. S., da Costa R.A.A.F., da Silva A.L.N., Rocha M.C.G., Coutinho F.M.B., Study of blends of HDPE and different grades of $CaCO_3$. Part 1. Factorial experimental design. Polym. Test, 2005, 24, 983–987.
28. Trawinski H., Hydocyclones, 2003, *in*: Ullmann's Encyclopedia of Industrial Chemistry (eds. M. Bohnet, C.J. Brinker, B. Cornils *et al.*). Wiley-VCH, Weinheim, vol 17, pp. 77–84.
29. Tukiendorf M., Krótkiewicz M., Boss J., The application of image analysis as a method of evaluation of granular blend's quality. Pol. J. Food Nutr. Sci., 2003, 22/53, No. 2, 27–30
30. Xu Y., Pelton R., A new look at how fines influence the strength of filled papers. J. Pulp. Pap. Sci., 2005, 31, 147–152.

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