

Application of Computer Image Analysis for Evaluation and Control of the Sucrose Crystallisation Process*

ZDENEK BUBNÍK, PAVEL KADLEC, HELENA ŠTARHOVÁ, VLADIMÍR POUR and ANDREA HINKOVÁ

Institute of Chemical Technology – Department of Carbohydrate Chemistry and Technology, Prague, Czech Republic

Abstract

BUBNÍK Z., KADLEC P., ŠTARHOVÁ H., POUR V., HINKOVÁ A. (2000): **Application of computer image analysis for evaluation and control of the sucrose crystallization process.** Czech J. Food Sci., 18: 23–28.

The paper deals with applications of computer image analysis to evaluation and control of crystallisation process in technical sugar solutions. Fields of application of this system: The kinetics study of the cooling crystallisation of sucrose from technical sugar solutions, measurement of the solubility of sucrose in technical sugar juice by the saturescopy method, measurement of crystal size distribution.

Key words: computer image analysis; saturescopic method; CSD; raw sugar juice

The quality of sugar juices obtained from natural resources is very variable and that is why for superior operation of crystallisation process it is necessary to measure the solubility and the process kinetics and according to obtained data to make corrections of operating programme parameters. Contemporaneous methods do not give the data rapidly and easily and so new methods are looked for.

The system of computer image analysis enables to extend the work at the crystallisation station and to make it substantially more efficient, which involves the evaluation of the produced crystals and the acquirement of the necessary substance properties and kinetic parameters.

The imaging system LUCIA G (= Laboratory Universal Computer Image Analysis) applied in this work is a product of the Laboratory Imaging Co., Prague, Czech Republic (ANON 1998). The firm offers both general purpose image processing and analysis systems and reliable customised solutions for scientific, biomedical, forensic and industrial imaging. LUCIA G is the true colour version. Grabbing, processing and analysis of images are performed at RGB or HSI colour space. Dedicated mainly for a 3-chip RGB TV camera. Supporting operating systems are Windows 3.x, Windows 95, 98, Windows NT 4.0, supporting grabbers are Matrox Magic, Matrox Comet and VGA. LUCIA includes a powerful, full-featured macro language and to-

gether with a rich image analysis library provides an excellent developer's environment for image analysis.

A wide spectrum of different objectives, microscopes, cameras and lighting systems enables to measure particles with different size range from several centimetres to microns. The program contains a large amount of tools for adjustment and arrangement of the image into the form suitable for optimal computer analysis.

The system enables easy calibration too. Except the different length parameters it is also possible to measure object areas, equivalent volumes, angles and many other parameters. The firm LAB-IMAGE Prague has developed a new software for easy manipulation and partial automatization of the whole process.

The paper deals with applications of computer image analysis to evaluation and control of crystallisation process in technical sugar solutions. Fields of application of this system:

- a) The kinetics study of the cooling crystallisation of sucrose from technical sugar solutions.
- b) Measurement of the solubility of sucrose in technical sugar juice – suitable application with the improvement of the saturescopy method.
- c) Measurement of distribution of the crystals obtained by the cooling crystallisation of raw sugar juice, evaluation of the crystal quality, colouring, size etc.

*This work was supported by grant project COPERNICUS SUCLEAN (No. ERBIC 15-CT96-0734).

Solubility Determination

The main part of the work deals with the solubility determination by a saturoscopic method because the newly-designed technology for sugar production (KLEMES *et al.* 1997) requires new analytical and controlling tools. The contemporaneous method for the determination of solubility by crystallisation experiment (when the equilibrium is achieved by crystallisation, BUBNÍK *et al.* 1997) is not operative and that is why the saturoscopic method seems to be a good solution (KADLEC 1971; VACCARI *et al.* 1993).

The influence of composition of impurities in technical sugar solutions on the solubility is shown in Fig. 1. This figure also depicts the course of sucrose solubility at the temperature 60°C at the different impurity contents. Other curves for different juices taken both from a standard sugar technology ("standard syrup") and a new technology based on the crystallisation of raw juice are also shown.

Saturoscopic instrument is a special microscope equipped with a heated plate. Supersaturated solution with the volume of several drops and containing microcrystals or particles of crushed crystals is placed between two thin glasses on the heating plate. The temperature linearly increases at a given rate.

The temperature at which the crystals start melting is detected as a temperature of saturation. The detection of this equilibrium point is very demanding for the experience of operating personnel and it is also the main disadvantage of the saturoscopic method. The Computer Image Analysis application significantly increases the measurement objectivity.

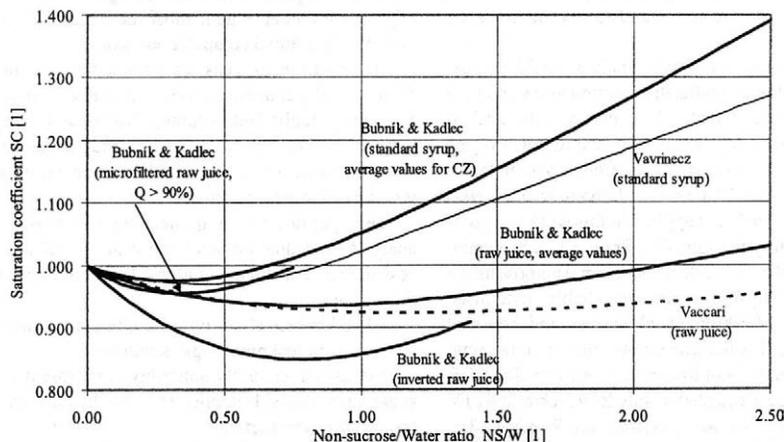
The firm LAB-IMAGE offers a special programming software for developing the programmes for the automatic temperature data reading, saving and evaluating of image series. The equilibrium point (saturation) achievement can be estimated by the operating personnel from a series of images. Another detection possibility is an evaluation of optical changes in chosen picture parts, e.g., a part of the observed crystal surface.

For the comparison of both methods the solubility of sucrose in the juices was also determined through crystallisation tests (BUBNÍK *et al.* 1997). The saturation temperature measurement by crystallisation and saturoscopic method was compared in 44 technical sugar solution samples. The statistical analysis (*t*-test) of the data shows that the difference between both methods at the level $\alpha = 0,05$ is not statistically significant.

Sucrose solubility in raw sugar beet juice was evaluated in the temperature interval 40–80°C and at the impurity content 0.2–1.8 (g/g) water. The results were used for evaluation of two-parametric equations (BUBNÍK *et al.* 1998a, b). The results are also shown in Fig. 1.

Crystal Size Distribution and Quality

The Computer Image Analysis is one of the most frequently used methods for granulometric particle measurement. Use of this method for crystal size evaluation in different samples can be considered to be almost a standard method. The LUCIA system offers great possibilities for image analysis. To get a desirable result you can change many parameters, e.g., length, width, max. and min. feret,



Saturation coefficient SC – ratio of sucrose solubility in technical solution and in water; Non-sucrose/Water ratio NS/W – ratio between impurity and water mass. Used data: experimental results and papers of BUBNÍK, KADLEC (1992) and BUBNÍK *et al.* (1997a, b), VACCARI *et al.* (1993) and VAVRINECZ (1978/79)

Fig. 1. The dependence of sucrose solubility in technical sugar solutions on the different impurity content at the temperature 60°C

e) All tables, charts and figures are possible to save, export and/or treat according to requirements.

f) All steps which were used during image analysis are saved and can be used for simple subroutine creating. This subroutine can be recalled and used to an analysis during similar samples measurement.

This system was applied to many measurements of particles in the food and chemical industry. The large sets of crystal samples from the cooling sugar crystallisation were analysed during the crystal size evaluation. The samples were measured both directly in a suspension and as a dry product after the separation and washing.

Important practical results were obtained during measurements of particle distribution in a slurry, which is a suspension of micro-crystals in alcohol solution. In the sugar plant the slurry is used for supersaturated sugar solution nucleation.

The analysis of crystals obtained by direct crystallisation of sucrose from raw sugar beet juice is shown as an example of measurement. This crystallisation method designed by VACCARI and MANTOVANI (1995) is a part of COPERNICUS project (KLEMES *et al.* 1997). Other results for cooling crystallisation that was running according to a simultaneous program and under the conditions: the temperature fall from 80 to 40°C, supersaturation 1.10 and impurity concentration 0.8 (g/g) water. Fig. 3 shows analysed crystals from one measured set, Fig. 4 summarises all analysed and calculated data (an output from the program LUCIA) and a histogram of crystal distribution is presented in Fig. 5.

Another important aim for LUCIA Image Analysis utilisation is a process application in sugar plants. In the following sugar campaign the system will be tested in an industrial crystallizer in the sugar plant. The main idea is to

verify the possibility of on-line measurements of crystal distribution.

Crystal Growth Kinetics and Nucleation

For crystal growth size measurement and for metastable zone determination a modified cell was employed which is usually used for cooling the saturoscope heating desk. The biggest advantages of this modified cell are the following parameters:

- Possibility of temperature maintenance.
- Low solution or suspension volume.
- It is possible to place this cell into the working space of an Image Analysis instrument.

The crystallisation runs under the conditions where a small number of crystals is growing in an surplus of mother liquor. The Image Analysis enables to evaluate different crystal parameters (Fig. 4). The time intervals are also possible to set by the program and the computer automatically records and saves images. It is possible to analyse all images after the crystallisation process.

Crystallisation rate is possible to determine either at the constant or changing temperature. The maintenance of temperature in a small amount of measured suspension allows to precisely operate the temperature and to apply the different cooling crystallisation methods in this way. The temperature is controlled by a computer with an equipment developed by BUBNIK *et al.* (1997). The course of temperature curve during the cooling crystallisation is calculated by a programme of simulation which was developed for sugar technical solution cooling crystallisation by BUBNIK and KADLEC (1994, 1996).

The disadvantage of this process is that the crystallisation runs without crystal movement in a mother liquor. A

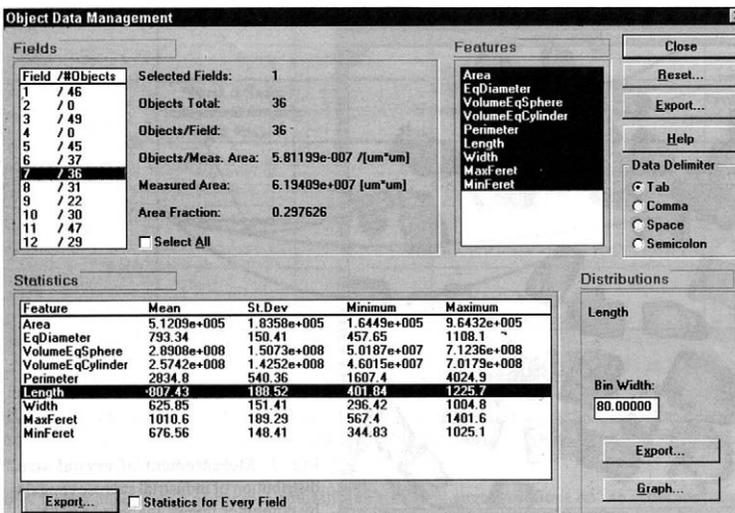


Fig. 4. Results of CSD (= Crystal Size Distribution) measurements by image analysis

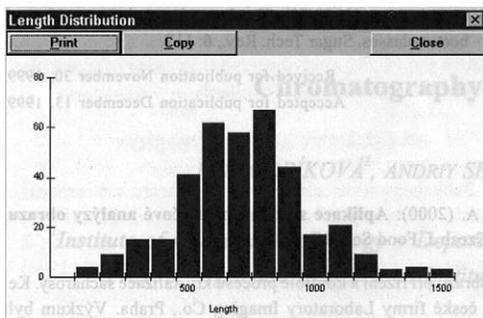


Fig. 5. Histogram of data from Fig. 4

new measuring cell with higher solution volume and equipped with the stirrer will be designed for the next measurement. But in the measuring period it will be necessary to switch off the stirring and let the crystals sediment. The innovating Image Analysis LUCIA system which was presented by the manufacturers in 1999 allows to scan the crystals during slow movement.

The system is being tested with the technical raw sugar juice solution. Kinetics of the crystal growth was measured by cooling crystallisation under the conditions complying with the newly-designed technological process in sugar production (project COPERNICUS – KLEMES *et al.* 1997). The conditions under which the crystallisation experiments were running result from technological process parameters: the temperature fall from 80 to 40°C, supersaturation 1.02–1.25 and impurity concentration 0.2–1.8 (g/g) water.

The tests which were carried out in this work followed above all the methodology verifying. Obtained kinetics data were used to complete the values measured by VACCARI *et al.* (1996) and to calculate equations describing sucrose crystal growth. The equations were input into the programmes simulating the course of cooling crystallisation (BUBNÍK *et al.* 1998b, 1999).

First tests for metastable zone width measurement were performed by cooling and showed the possibility to apply this method for nucleation study. With a suitable set of lighting (e.g., side lighting with glass fibres) the optical properties change at the point where the new phase rises and it is easy to detect the forming of new particles.

Conclusion

A saturoscopic method for the solubility measurement of high viscous solution with wide metastable zone was suggested and successfully realised in this paper. In further work the system will be verified for different kinds of solutions and also other tests will be carried out directly in the industrial plant.

A procedure for direct observation and evaluation of growing crystals under the isotherm conditions and during the controlled cooling crystallisation was suggested.

The system was successfully applied to the crystal size distribution measurement and also to many other particles from the products of chemical and food industry. Also a methodology for the size evaluation of sucrose crystals obtained from different types of industrial crystallises and from laboratory tests was worked out.

In further research the possibilities of LUCIA Image Analysis application will be verified in industrial conditions in the sugar crystallisation process. The aim is to test the possibility of on-line measurement of the crystal distribution in industrial operations.

References

- ANON (1998): LUCIA G – Laboratory Universal Computer Image Analysis. Laboratory Imaging Co., Prague, Czech Republic. e-mail: lim@lim.cz, web site: www.lim.cz.
- BUBNÍK Z., KADLEC P. (1992): Solubility of sucrose in impure sugar solutions. *Zuckerindustrie*, **117**: 619–625.
- BUBNÍK Z., KADLEC P. (1994): Modelling and Dynamic Simulation of Cooling Crystallisation of Sucrose. In: BIMBENET J. J. *et al.* (Eds.): *Automatic Control of Food and Biological Processes*. Elsevier, Amsterdam: 175–182.
- BUBNÍK Z., KADLEC P. (1996): Program for Simulation of Cooling Crystallisation of Sucrose. In: Proc. 20th General Assembly C.I.T.S., Munich 1995, Dr. A. Bartens Pub. Co., Berlin: 30–39.
- BUBNÍK Z., KADLEC P. (1998a): Comparison between the saccharose crystal growth in impure sugar juices and that in juices treated with ultrafiltration. In: Proc. 13th Congr. CHISA'98 (CD ROM), Prague.
- BUBNÍK Z., KADLEC P. (1998b): Solubility and crystal growth of sucrose from raw sugar beet juices. In: Congr. ICCG-12/ICVGE-10, Jerusalem, Israel.
- BUBNÍK Z., KADLEC P., STARHOVÁ H., HINKOVÁ A., POUR V. (1999): Sucrose crystal growth from concentrated raw juice. In: 21th General Assembly C.I.T.S., Antwerp.
- BUBNÍK Z., STEFFEK P., STARHOVÁ H., KADLEC P., UHEREK M., POUR V. (1997): Solubility of sucrose in sugar raw juice. *Listy Cukrov. Řep.*, **113**: 266–271.
- KADLEC P. (1971): Stanovení koeficientu přesycení cukerných roztoků saturoskopem. *Listy Cukrov.*, **87**: 164–167.
- KLEMES J., URBANEC K., VACCARI G., MANTOVANI G., BUBNÍK Z., LENTINI A., KADLEC P., PLACEK I. (1997): Minimisation of energy and water use in sugar production by clean technology based on cooling crystallisation of concentrated raw juice – SUCLEAN. The EC COPERNICUS Project ERBIC 15-CT96-0734.
- VACCARI G., MANTOVANI G. (1995): A pilot plant for continuous crystallisation from raw juice. *Int. Sugar J.*, **97**: 209–218.
- VACCARI G., MANTOVANI G., SGUALDINO G., ANDREOLI F. (1993): Cooling crystallisation of raw juice – Determination of sucrose solubility. *Zuckerindustrie*, **118**: 780–783.

- VACCARI G., MANTOVANI G., SGUALDINO G., DOSI E. (1996): Cooling crystallisation of raw juice. Zuckerindustrie, **121**: 111–117.
- VAVRINECZ G. (1978/79): The formation and composition of beet molasses. Sugar Tech. Rev., **6**: 117.

Received for publication November 30, 1999

Accepted for publication December 13, 1999

Souhrn

BUBNÍK Z., KADLEC P., ŠTARHOVÁ H., POUR V., HINKOVÁ A. (2000): Aplikace systému počítačové analýzy obrazu LUCIA při řízení a kontrole procesu krystalizace sacharosu. Czech J. Food Sci., **18**: 23–28.

Práce se zabývá možnostmi aplikace systému počítačové analýzy obrazu při řízení a kontrole procesu krystalizace sacharosu. Ke studiu byl využit systém počítačové analýzy obrazu LUCIA G české firmy Laboratory Imaging Co., Praha. Výzkum byl prováděn v rámci grantového projektu COPERNICUS SUCLEAN, který je zaměřen na snížení spotřeby energie a vody pomocí aplikace nové technologie založené na krystalizaci surové řepné šťávy. Složení surové šťávy je ve srovnání s cukernými šťávami vyrobenými standardní technologií mnohem variabilnější, proto je nutné hledat nové analytické a řídicí metody, jež by umožnily operativně zasahovat do technologického procesu. Počítačová analýza obrazu se jeví jako nadějná metoda splňující uvedené požadavky. Při řešení projektu byly provedeny následující aplikace systému LUCIA: a) měření rozpustnosti sacharosu v surové cukerné šťávě pomocí saturoskopické metody; b) studium kinetiky chladičích krystalizace sacharosu ze zahuštěné surové šťávy a stanovení šířky metastabilní oblasti, což je nutné pro simulaci i řízení krystalizačního procesu; c) měření distribuce krystalů získaných chladičích krystalizací surové cukerné šťávy, hodnocení jejich kvality, zbarvení, velikosti apod. Získané výsledky včetně vyvinutého software a hardware potvrdily přednosti počítačové analýzy a navržené metody je možné využívat v laboratorních i průmyslových podmínkách.

Klíčová slova: počítačová analýza obrazu; saturoskopická metoda; surová cukerná šťáva

Corresponding author:

Doc. Ing. ZDENĚK BUBNÍK, CSc., Vysoká škola chemicko-technologická, Ústav chemie a technologie sacharidů, Technická 5, 166 28 Praha 6, Česká republika, tel.: + 420 2 24 35 31 12, fax: + 420 2 311 99 90, e-mail: zdenek.bubnik@vscht.cz
