

SIMULATION OF VACUUM CRYSTALLIZER

A DISSERTATION

*Submitted in partial fulfillment of the
requirements for the award of the degree*

of

MASTER OF TECHNOLOGY

in

CHEMICAL ENGINEERING

(With Specialization in Computer Aided Process Plant Design)

By

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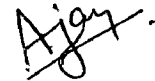
Candidate's Declaration

I hereby declare that the report which is being presented in this project work "SIMULATION OF VACUUM CRYSTALLIZER" in partial fulfillment of the requirements for the award of the degree of **Master of Technology** in Computer Aided Process Plant Design, submitted in Chemical Engineering Department, Indian Institute of Technology, Roorkee is an authentic record of my own work carried out during a period from June 2005 to June 2006 under the supervision of **Dr. V.K. Agarwal**, Chemical Engineering Department, Indian Institute of Technology, Roorkee.

I have not submitted the matter embodied in this report for the award of any other degree or diploma.

Date: 28 June, 2006

Place: Roorkee



(AJAY BANSAL)

This is to certify that the above statement made by the candidate is correct to the best of my knowledge.



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ABSTRACT

The present investigation pertains to a theoretical study on the simulation of vacuum crystallizer of sugar industry. It basically deals with the development of a mathematical model for crystallizer. It also includes the solution technique for the solution of the model. In the end, studies of the effect of different operating parameters viz. crystal content, crystal mean aperture, purity of massecuite and brix of massecuite on the model behaviour has been done. The steady state mathematical model is based on flow pattern.

Using the basic principles of mass conservation and energy conservation a mathematical model of a vacuum crystallizer has been developed. These balance equations include material balance and enthalpy balance equations. Correlations for enthalpies of syrup and massecuite have been developed. The resulting system of equations is solved using "Iterative method". The steady state mathematical model developed describes the most important aspects of the crystallizer behaviour in each compartment: crystal content, crystal mean size

The results of the simulation have been compared with those due to Bounahmidi (2001). An examination of the results shows, that the model has been successful in representing the vacuum crystallizer. The maximum deviation between the profiles obtained by this simulation with those due to Bounahmidi (2001) was found to be less than 4%.

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It is with great pleasure that I take this opportunity to bow my head in respect and gratitude for all those who helped me in making this project a great success. I am in dearth of words to express myself in such a joyous moment.

I take this opportunity to grace myself from the benign self of my teacher and guide, Dr.V.K.Agarwal, for ushering me from theoreticality to practicality and from plutonic to pragmatic ideas. No rhapsody or rhetoric eloquence can replace of what he had done for me and the way he has helped me in bringing out this project. I will always be indebted to him all our long life.

I would also like to thank Dr. Shri Chand, Professor and head, Chemical Engineering Department, for providing various facilities during this dissertation.

I also like to thank my friends for patiently bearing me and helping me in what ever way possible through their expert advices and encouraging me to go deeper in my project.

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NOMENCLATURE

Bx	Brix: mass fraction of dry substances in total suspension	(wt %)
C	Mass of crystals	(kg)
Cr	Crystal content: mass fraction of crystals in total suspension	(wt %)
F	Mass flow rate	(kg/h)
G	Linear rate	(m/h)
h	Specific enthalpy	(kJ/kg)
h _v	Latent heat of water vaporization	(kJ/kg)
I	Impurities in the molasses in pan	(kg)
K _a	Surface shape factor	
K _v	Volume shape factor	
MA	Crystal mean aperture	(m)
N	Number of crystals	
OS	Oversaturation of molasses	
OS _{crit}	Critical Oversaturation	
P	Purity: mass fraction of sucrose in dry substances	(wt %)
r _c	Mass rate	(kg/m ² h)
S	Sucrose content in the molasses in pan	(kg)
SC	Solubility coefficient	
Sc	Heat transfer surface area	(m ²)
Scr	Total surface area of crystals	(m ²)
SS	Supersaturation of sucrose in molasses	
T	Temperature	(°C)
U	Heat transfer coefficient	(kJ/h m ² °C)
W	Water in the molasses in pan	(kg)

INTRODUCTION

Currently, with the development and progress made in computer technology, modeling and simulation are very helpful tools for design, monitoring, performance analysis, control and optimization of industrial processes. The present work deals with applying these tools to the continuous crystallization unit of sugar manufacturing which constitutes an important industrial activity.

A simplified description of the sugar manufacturing process is given in Fig. 1. The process concerns standard raw cane sugar manufacturing without refining. The first step consists of juice extraction by crushing and grinding the cane sugar. The product, which is a dark-green juice, is acid and turbid and needs purification to remove the maximum of impurities it contains. This is achieved through lime treatment followed by filtration. The purified juice is concentrated into syrup by vacuum multiple-effect evaporation unit. The obtained syrup is then crystallized to obtain raw sugar as a final product and molasses as a residual solution after centrifugation.

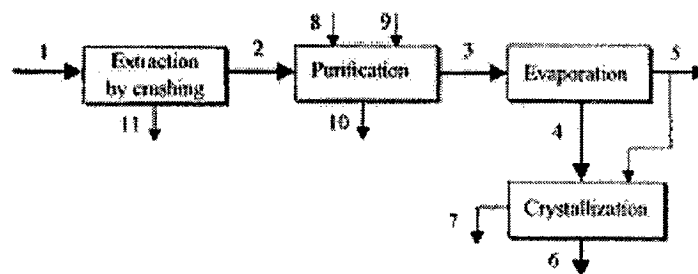


Fig 1.1: Schematic flow diagram of raw cane sugar manufacturing. (1)Cane sugar; (2) mixed raw juice; (3) clear juice; (4) syrup; (5) heating steam; (6) raw sugar; (7) molasses; (8) milk of lime (CaO); (9) H₃PO₄; (10) scums; (11) bagass.

The crystallization unit, shown in Fig. 2, is composed of three vacuum continuous crystallizers (pans). The first boiling of raw syrup leads to a first grade massecuite (A), which yields raw sugar and a molasses residue. This residue is returned to the second pan to be reboiled to a second massecuite (B) that yields B sugar of less purity. The same operation is carried out in the third boiling to obtain C sugar from the low-grade

massecuite (C). Sugars B and C are, respectively recycled as magma to the 1st and 2nd pan. Purity of sugar crystals produced decreases from the first to the third crystallizer.

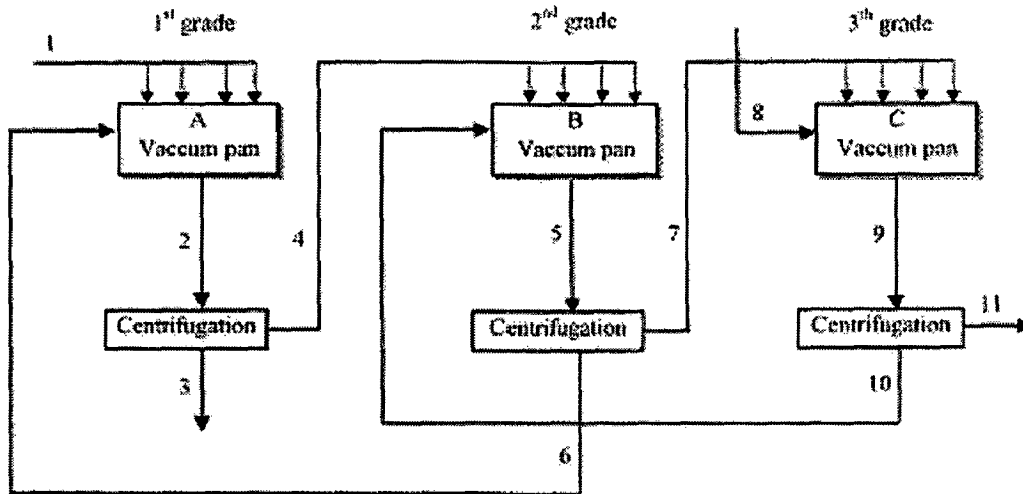


Fig 1.2: Schematic representation of sugar crystallization unit. (1) Syrup (liquor A); (2) massecuite A; (3) raw sugar (A); (4) mother liquor A used as feed liquor B; (5) massecuite B; (6) sugar B recycled as magma A; (7) mother liquor B used as feed liquor C; (8) magma C (prepared in batch seeding crystallizer); (9) massecuite C; (10) sugar C recycled as magma B; (11) molasses.

The economy of sugar manufacturing process hinges on the extraction of maximum sugar and producing crystals having the highest purity and uniform size. This mainly depends on the design and performance of the purification and the crystallization units that play a decisive role regarding the quality of the product and efficiency of the whole process.

Sugar crystallization operation is well known as a complex process including a large number of interacting operating variables. Modeling and simulation of this process are powerful means to find optimal operating conditions of crystallizers and hence ensure good performance. Good performance requires control of the supersaturation profile, which is the most important parameter for crystallization process. Unfortunately, reliable devices allowing direct measurements of this parameter are not yet available.

It is well-known that crystal growth phenomenon in the sugar crystallization process of the sugar industry is complex due to large number of interacting process variables. Moreover, at present sugar crystallization is a sequential batch process where the text operation could be started only after the preceding operation is satisfactorily completed.

As a general rule, uniform size crystal growth takes place only in the metastable zone of the supersaturation level which must be maintained throughout the boiling strike. It is observed that the degree of supersaturation is a strong function of temperature and therefore on the pan vacuum and also on steam pressure which are the two main intakes to the process.

In order to ensure uniform growth of the sugar crystal of dimensions of the order of 0.2 to 0.3 mm starting from a grain of 3 to 5 μm size in the minimum possible time with minimum energy consumption without forming conglomerates, it is necessary to control the rate of evaporation with steady conditions of supersaturation. To be able to maintain steady conditions of supersaturation the input feed rate of molasses is to be so regulated in conjunction with the heating media viz. steam flow rate, that uniform evaporation can be maintained for most of the time in the crystallization process. This in other words, means that the water evaporated due to boiling is counterbalanced by the water which is coming as input along with molasses and therefore leading to continuous transfer of sugar from the liquid phase to the solid phase so that the crystal size increases accordingly.

1.1 CRYSTALLIZATION

Crystallization is the formation of solid particles within a homogeneous phase. It may occur as the formation of solid particles in a vapor, as in snow; as solidification from a liquid melt, as in the manufacture of large single crystals; or as crystallization from liquid solution. Crystallization is also a solid-liquid separation process in which mass transfer occurs of a solute from the liquid solution to a pure solid crystalline phase. An important example is in the production of sucrose from sugar beet, where the sucrose is crystallized out from an aqueous solution. This process can occur in the freezing of water to form ice and in the formation of solid crystals from a liquid solution.

In crystallization the solution is concentrated and usually cooled until the solute concentration becomes greater than its solubility at that temperature. Then the solute comes out of the solution forming crystals of approximately pure solute.

In industrial crystallization from solution, the two phase mixture of mother liquor and crystals of all sizes, which occupies the crystallizer and is withdrawn as a product, is called *magma*.

In commercial crystallization the yield and purity of crystals are not only important but also the size and shapes of crystals. It is often desirable that crystals be uniform in size. Size uniformity is desirable to minimizing caking in the package, for ease of pouring, for ease in washing and filtering, and for uniform behavior when used. Sometimes large crystals are requested from the purchasers, even though the smaller crystals are just useful. Also, crystals of a certain shape are sometimes required, such as needles rather than cubes. For these reasons, crystal size distribution (CSD) must be under control; it is a prime objective in the design and operation of crystallizers.

1.2 MECHANISM OF CRYSTALLIZATION

The attainment of the supersaturated state is essential for any crystallization operation, and the degree of supersaturation, or deviation from the equilibrium saturated condition, is the prime factor controlling the deposition process. Any crystallization operation can be considered to comprise three basic steps:

1. Achievement of supersaturation or supercooling.
2. Formation of crystal nuclei.
3. Growth of the crystal.

All three processes may be occurring simultaneously in different regions of a crystallization unit. The ideal crystallization of course, would consist of a strictly controlled step-wise procedure, but the complete cessation of nucleation cannot normally be guaranteed in a growing mass of suspended and circulating crystals. The supersaturation of a system may be achieved by cooling, evaporation, the addition of a precipitant or as a result of the chemical reaction between two homogeneous phases.

1.2.1 NUCLEATION

The rate of nucleation is the number of new particles formed per unit time per unit volume of magma or solids-free mother liquor. This quantity is the first kinetic parameter controlling the CSD. The condition of supersaturation alone is not sufficient cause for a

system to begin to crystallize. Before crystals can grow there must exist in the solution a number of minute solid bodies known as centers of crystallization, seeds, embryos or nuclei. Nucleation may occur spontaneously or it may be induced artificially; these two cases are frequently referred to as homogeneous and heterogeneous nucleation, respectively. It is not always possible, however, to decide whether a system has nucleated of its own accord or whether it has done so under the influence of some external stimulus.

The term 'primary' will be reserved for all cases of nucleation, homogeneous or heterogeneous, in systems that do not contain crystalline matter. On the other hand, nuclei are often generated in the vicinity of crystals present in a supersaturated system; this phenomenon will be referred to as 'secondary' nucleation.

1.2.2 CRYSTAL GROWTH

The rate of growth of a crystal face is the distance moved per unit time in a direction that is perpendicular to the face. The crystal growth is a layer by layer process, and since the growth can occur only at the outer face of the crystal, the solute material must be transported to that face from the bulk of the solution. The solute molecules reach the face by diffusion through the liquid phase. The usual mass transfer coefficient k_y applies in this case. At the surface the distance to integration of the molecules into the space lattice at the face must be considered. This reaction at the surface occurs at a finite rate, and the overall process consists of two resistances in series. The solution must be supersaturated for the diffusion and interfacial steps to proceed.

1.2.3 THE ΔL LAW OF CRYSTAL GROWTH

McCabe has shown that all crystals those are geometrically similar and of the same material in the same solution grow at the same rate. The growth is measured as the increase in length ΔL , in mm, in linear dimension of the crystal. This increase in length is for geometrically corresponding distances on all crystals. This increase is independent of the initial size of the initial crystals, provided that the crystals are subject to the same environmental conditions. This law follows, where the overall transfer coefficient is same for each face of all crystals.

Mathematically, this can be written

$$\Delta L/\Delta t = G$$

where Δt is time in h and growth rate G is a constant in mm/h. hence, if D_1 is the line dimension of a given crystal at time t_1 and D_2 at time t_2 ,

$$\Delta L = D_2 - D_1 = G (t_2 - t_1)$$

The total growth ($D_2 - D_1$) or ΔL is the same for all crystals.

The ΔL law fails in cases where the crystals are given any different treatment on size. It has been found to hold for many materials, particularly when the crystals are under 50 mesh in size (0.3 mm). This law is not applicable in all cases, and reasonably accurate in many situations.

1.3 VACUUM CRYSTALLIZER

The term 'vacuum' crystallization is capable of being interpreted in many ways; any crystallizer that is operated under reduced could be called a vacuum crystallizer. Some of the evaporators described above could be classified in this manner, but these units are better, and more correctly, described as reduced-pressure evaporating crystallizers. The true vacuum crystallizer operates on a slightly different principle: supersaturation is achieved by simultaneous evaporation and adiabatic cooling of the feed solution. These units, therefore, act as both evaporators and coolers.

To demonstrate the operating principles of these units, consider a hot saturated solution introduced into a lagged vessel maintained under vacuum. If the feed temperature is higher than that at which the solution would boil under the low pressure existing in the vessel, the feed solution cools adiabatically to this temperature. The sensible heat liberated by the solution, together with any heat of crystallization liberated owing to the deposition of crystals at the lower temperature, causes the evaporation of a small amount of the solvent, which in turn results in the deposition of more crystals owing to the increased concentration.

In a continuously operated vacuum crystallizer the feed solution should reach the surface of the liquor in the vessel quickly, otherwise evaporation and cooling will not take place, because, owing to the hydrostatic head of solution, the boiling point elevation becomes appreciable at the low pressures (7-20 mbar, 5-15 mm Hg) used in

these vessels, and the feed solution will tend to migrate down towards the bottom outlet. Care must be taken, therefore either to introduce the feed near the surface of the liquor in the vessel or to provide some form of agitation.

1.4 OBJECTIVE OF DISSERTATION

This work has been planned with the following objectives.

1. To develop a model, of sugar continuous vacuum crystallizer. The mathematical model is based on a flow pattern.
2. Simulation of the model using numerical technique.
3. To check and validate the model against plant measurements.
4. To check the effects of the various parameters on the model and the operation of the crystallizer.

LITERATURE REVIEW

Kapur, P., Patil, V.L., Gautam, G.K. and Acharya, G.N. (1987)

This paper describes the theoretical studies made towards the development of a mathematical model of sugar crystallization process through its basic equations of mass and energy balance. The controller structure is so determined that it exhibits a decoupled performance of the process under varying input conditions. It is observed by the author that the degree of supersaturation is a strong function of temperature and therefore on the pan vacuum and also on steam pressure which are two main intakes to the process.

The basic equations of mass and energy balance are derived making the following assumptions:

- (i) Crystallization take place under high purity syrups having no impurity.
- (ii) Masecuite evaporates at saturation temperature.
- (iii) Vapour evacuation takes place at saturation level.
- (iv) Incoming steam is under constant-pressure, temperature and hence has constant enthalpy.
- (v) Crystallization heat is negligible as compared to that of masecuite.
- (vi) Incoming feed molasses has constant concentration, density, temperature and also constant enthalpy.
- (vii) Pan area is very large as compared to the incremental change in the pan level due to crystal growth.
- (viii) Total heat capacities are fixed at their average values.

The process dynamics have been expressed both in frequency and in time domain and simulated on Intel MDS 287 FD Microprocessor Development System in PASCAL programming language.

Liu, C.H., Zhang, D.H., Sun, C.G. and Shen, Z.Q. (1991)

Based on the population balance, a model for simulating a practical industrial crystallizer with 12 stages has been developed. The model parameters have been

estimated by fitting the operating data which were obtained from measuring the samples withdrawn from stages of crystallizer. Using the model with these parameters, the effect of operating conditions on crystal size distribution in each stage of this unit has been determined and the optimum condition has been established.

The crystallization kinetic parameters in this model have been estimated by analyzing the supersaturation of the liquor and the crystal size distribution of the samples which were withdrawn from several crystallization chambers under various operation conditions in this apparatus. Model is developed for the production of adipic acid.

The simulated results reveal clearly the effects of operating conditions on the quality of the product crystal, so that the optimum operating conditions established by analyzing the results of simulation can be used to direct and modify the operation of this apparatus.

Gros, D.H. (1979)

In this paper a deterministic model of batch evaporative sugar crystallization is developed. This model accounts for all four phases of normal pan boiling performance but it does not include heat transfer considerations. Furthermore, the choice of dependent and independent variables as well as necessary empirical correlations are discussed. The model is built in order to enable rigorous simulation of industrial vacuum pan crystallizer.

The mathematical model to be presented in this paper has these assumptions:

- (i) No nucleation and no dissolving of crystals.
- (ii) Complete back mixing conditions.
- (iii) Constant percentage of non-sugar in crystalline phase.

In practice conditions 1 and 2 are approximately fulfilled through efficient impeller mixing and careful boiling with in the metastable range. Assumption 3 is not altogether correct because the percentual non-sugar in the mother liquor at the end of this strike.

Semlali, N., Hassani, A., Saidi, K. and Bounahmidi, T. (2001)

In this paper the model is based on flow pattern, which was assumed and validated against plant measurements using a tracer test. The steady state mathematical model developed describes the most important aspects of the crystallizer behavior in each compartment: supersaturation, crystal size distribution and flow rate of the product crystals. The model can also describe the undesirable behavior such as dissolution and nucleation. Validation of the developed model was performed using industrial data. A parametric sensitivity study confirmed that the syrup supply distribution is the main variable that should be manipulated to achieve good performance for the crystallizer.

The mathematical model proposed in this work to describe the steady state behavior of sugar crystallizer is based on mass, energy and population balance equations. It is set up on the following simplifying assumptions:

- (i) Crystal growth rate is assumed to be size independent. This is a reasonable assumption because size dependent phenomenon is not apparent in sugar crystallization as it is reported by many workers.
- (ii) Impurities (non-sugars) are transferred from the solution to the crystal such that crystal purity remains constant during the operation.
- (iii) Attrition, crystal breakage and agglomeration phenomena are generally neglected in crystallization. This approximation is justified in the case of sugar crystallization because of the low shear involved and because of the fact that agglomeration is not usually significant for sucrose.
- (iv) Because of the very small value of its flow rate, the superheated steam used for mixing the sugar suspension was neglected.

The Newton-Raphson was used for solving the equations of the mathematical model. This model enables the evolution of the main operating parameters such as the supersaturation profile, the crystal size distribution and the flow rate of the sugar crystals produced to be described. It was confirmed that the lateral syrup distribution is the main variable that should be manipulated in order to achieve good control of supersaturation profile and thus good performance of plant crystallization.

Georgieva, P., Meireles, M.J. and Foyo de Azevedo, S. (2003)

The application of knowledge-based hybrid modeling to an industrial scale batch evaporative crystallization Process in cane sugar refining is reported. First, principles models of the process lead in general to good description of process state, expect for the prediction of the main crystal size distribution parameters mean size and coefficient of variation. This is caused by difficulties in expressing accurately nucleation and crystal growth rates and especially the complex phenomena of agglomeration in the relevant population balance. A hybrid model is proposed, which combines a partial mechanistic model that reflects the general mass, energy, and population balances with a neural network to express growth rate, nucleation kinetics, and agglomeration phenomena. Results demonstrate a better agreement between experimental data and hybrid model prediction than that observed with the complete mechanistic model.

Semlali, N., Hassani, A., Saidi, K. and Bounahmidi, T. (2002)

To achieve an automatic control of the crystallization unit of sugar factory and insure its good performance, a thorough understanding of the physicochemical phenomena involved is required. In order to contribute to this effort, a steady state phenomenal model was previously developed and validated, using plant data, for a 1st grade cane sugar continuous crystallizer. The testing of the validity of the developed model in order to study the performance of the 2nd and 3rd stages of a beat sugar crystallization unit is discussed. Plant data reconciliation was performed by exploiting data redundancy, using general data reconciliation method. Simulation results obtained were in satisfactory agreement with the reconciled plant data for the whole crystallization stages. The model is also able to describe the monitoring anomalies which can occur under an improper adjustment of operating conditions. On the other hand, this paper confirms a conclusion of an earlier paper that the lateral syrup supply distribution is the main variable that should be tuned to achieve high performance for the crystallization process.

Hassani, A., Semlali, N. and Bounahmidi, T. (2002)

This paper deals with development of steady state model in order to optimize the continuous pans of sugar crystallization workshops. For this objective, a mixing model was proposed and validated using plant measurements tracer test. Therefore, a mathematical model was developed. The model is able to describe the most important aspects of the physicochemical behavior of the crystallizer: supersaturation profile, crystal size distribution, and flow rate of the product crystals. This model takes into account the dissolution and nucleation phenomena. It was validated on an industrial continuous crystallizer in a Moroccan sugar factory in order to optimize its performance.

Maurandi, S. (1998)

Sucrose 3-step industrial crystallization scheme typical of north European sugar factories was evaluated using mathematical models. The important parameters in saturated sucrose solution boiling process (temperature, viscosity, crystallization speed, crystal seeding, mixing time, ebullioscopic value changes) were analyzed and interesting correlations between process variables were found. The most important crystallization parameters for practical sucrose manufacture and production yields are emphasized.

Depeyre, D., Isambert, A., Ducloux, P., Chraïbi, H., Daubois, P., Kuipers, S. and Roy, D. (1989)

In this paper dynamic simulation model was established and validated according to data registered in an operating plant. The model, which gave information on the evolution of the crystal size distribution, was used to test different control strategies. The chosen strategy was based on the control of super saturation, by action of the steam flow rate. Micro computer software was written and the system included management of the upstream and the down stream parts of the plant.

Bubnik, Z. and Kadlec, P. (1989)

In this paper a laboratory scale continuous crystallizer using isothermal evaporation at reduced pressure was designed and developed to model the vacuum pan

arrangement used in the sugar industry. The apparatus was automatically controlled the conductometric method and crystallization experiments were performed using the methods of crystal population density balance from molecular sieve analyzed data.

Guerra, D. and Jorge. (1984)

In this paper the theory of masseruite crystallization by cooling was analyzed, and the application of a mathematical model, based on crystallization rate data for the establishment of optimal cooling conditions, was described. A yield of sucrose crystals greater than 40% was predicted by this model at cooling rate $1/3^{\circ}\text{min}^{-1}$, which is in good agreement with industrial experience in vacuum pan crystallization.

Lekawski, W. and Bober, A. (1977)

In this paper a mathematical model was developed for the crystallization of sucrose in industrial installations. The model, which involved 42 parameters, was used to obtain an algorithm for the control of the crystallization process with the COMPUCORP 425 G mini computer system. This control system was used during the start up and initial operation of a new sugar refinery.

Todes, O.M. and Litunovskii, N.I. (1966)

By analyzing the linear growth rate of crystals under the assumption that the kinetics of crystallization in industrial applications is limited by the rate of molecular diffusion, the limiting condition for the Nusselt criterion was determined. Two examples are discussed: the crystallization of sugar from syrup through evaporation and the crystallization of $\text{K}_2\text{Cr}_2\text{O}_7$ from a solution cooled in a crystallizer. The optimal conditions are defined on a laboratory scale for the two methods for crystallization.

Doucet and Giddey (1966)

They were the first to publish a model for a batch sugar pan. The model they developed was a dynamic model for high and medium purity systems. The model used integral equations that were solved by analogue computation. The concept in their model is that the water evaporated from the pan must be balanced by two effects

(1) The sucrose removed from the pan molasses to maintain the over saturation at high enough value for crystallization.

(2) The water added from the feed required to replace the sucrose loss due to deposition

They considered impure solution, but did not model the crystal size distribution nor considered controlling the crystal content.

Evans et al. (1970)

They suggested a model for refinery pan operation. Their model included mass and energy balances and also intensive variables such as solution concentrations and crystal mass fraction. This work was done to analyze the sensitivity of sugar production to process variables and also to fit industrial data to the model.

Wright (1971)

Prior to the release of the model by Wright (1971), Wright and White (1968) published a paper on a mathematical simulation of the fed-batch impure sucrose crystallization system. The model included the following;

- Mass and energy balances
- Equilibrium phase relationships
- Crystal and nucleation rate expressions
- Dynamic crystal inventory, as done by Ciolan (1966)

It was made clear that most of the equations except the mass and energy balance may not be exact expressions.

The work done by Wright (1971) resulted in the development of the model for the batch pan that included the population balance dynamics as moment relations, crystal size distribution and the growth rate parameters for an industrial batch pan. He also did some industrial testing of the batch model.

Broadfoot (1980)

Broadfoot carried out designs on the continuous pan and then optimized the continuous pan for various operating conditions. His designs were based on a steady state model and contained moment's relations.

Wilson (1990)

Wilson dealt with the control of fed-batch impure sucrose crystallization in a vacuum pan. Wilson's task was the application of a non-linear control algorithm (Generic Model Control) to the vacuum pan. Wilson showed that given sufficient plant model mismatch, algorithm performance deteriorates to the point where the GMC was equivalent to the PID controller or in the worst case became unstable.

Wilson also attempted to implement a state estimator that would deliver the on-line predictions of the process states and their derived variables, such as the sucrose OS and the crystal content (CC). The estimator used by Wilson was based on the Extended Kalman filter (EFK), (Kalman, 1960) presented by Hamilton et al. (1973).

Schneider (1996)

Schneider developed further the work by Wilson and others by improving the controls for the fed-batch pans. He studied the addition of the state estimator (Schmidt-Kalman Filter or SEKF). This estimator was capable of accounting for the impact of uncertain process parameters and/or unobservable model states in such a way that they kept the other model states open to the process measurements employed for feedback to the model.

Rein, P.W., Echeverri, L.F. and Acharya S. (2004)

The crystallization of sucrose involves complex processes that require the correct design of vacuum pans and precise operation. Numerous parameters, such as tube dimensions, down take size and pan geometry, determine the quality of the sugar produced and the throughput, which results in certain vacuum pans giving better performance than others. A particularly important factor is the circulation, which is strongly interrelated with the convective-boiling heat transfer in the calandria and

determines, to a large extent, the velocity of crystallization and the uniformity of the conditions inside the vessel. In this paper several factors that affect boiling and circulation in vacuum pans are identified. The use of Computational Fluid Dynamics in the analysis of circulation and in the evaluation of alternatives for hydrodynamic optimization is discussed. Options for making changes to batch pans to improve the characteristics of the pan are identified, either to change the geometry or assist circulation. Some results with steam-assisted circulation (steam jiggers) are given, which show how the performance of a pan can be improved.

Lauret, P., Boyer, H. and Gatina, J.C. (2000)

The first and maybe the most important step in designing a model-based predictive controller is to develop a model that is as accurate as possible and that is valid under a wide range of operating conditions. The sugar boiling process is a strongly nonlinear and nonstationary process. The main process nonlinearities are represented by the crystal growth rate. This paper addresses the development of the crystal growth rate model according to two approaches. The first approach is classical and consists of determining the parameters of the empirical expressions of the growth rate through the use of a nonlinear programming optimization technique. The second is a novel modeling strategy that combines an artificial neural network (ANN) as an approximator of the growth rate with prior knowledge represented by the mass balance of sucrose crystals. The main objective of this paper is to demonstrate that NLP performs local fitting while the hybrid neural network gives rise to a more flexible model of the growth rate. Consequently, the scope of nonlinearities of concern is much wider. The first results show that the first type of model performs local fitting while the second offers a greater flexibility. The two models were developed with industrial data collected from a 60 m³ batch evaporative crystallizer.

Puel, F., Fevotte, G. and Klein, J.P. (2003)

In order to obtain constant solid properties with particles exhibiting a low order of symmetry, it is necessary to monitor and to control several distributed parameters characterizing the crystal shape and size. A bi-dimensional population balance model was

Dixon, T., Hobson, P., Mann, A., Plaza, F., Pennisi, S. and Steindl, R. (2003)

This paper summarizes a wide range of ongoing applications of CFD in the sugar industry. Principal among these have been the development of bagasse combustion technologies and understanding the processes involved in bagasse combustion and steam generation. SRI has been active for over 10 years in CFD with the FURNACE code, encompassing the full range of applications from fundamental code development, the generation and commercialization of new ideas and technologies, to the resolution of practical plant problems. Typical areas have included particle erosion, corrosion and heat transfer in tube bundle units, particle drying, ignition and burnout dynamics, the development of advanced boiler designs and more recently CO and NO_x pollutant generation and reduction.

CFD has been applied extensively in the design of the new-generation clarifier that has achieved throughput increases of over 75% with improved clarified juice quality. Other applications of CFD that are summarized include the modelling and design upgrading of evaporators, capacity and design improvements including stirrer retrofits to vacuum pans used in sugar crystallization, and the development of bagasse gasification technology for advanced power generation.

Essemiani, K., Traversay, C.D. and Gallot, J.C. (2004)

The CFD technique is used to describe the mixing conditions in a pilot-scale FCC (forced-circulation crystallizer) and to study the impact of flow rate and aspect ratio on local flow conditions and RTD (residence time distribution) in the crystallizer. The analysis adequately predicts the oscillating flow and two phase (gas-liquid) interaction at the free surface. A comparison has been made between the CFD predictions and models of RTD. The results support the use of CFD methodology as an aid to optimization of commercial-scale FCC design.

Jansens, P.J., Bruinsma, O.S.L. and Rosmalen, G.M.V. (1995)

Cascades of crystallizers are applied in many processes. The optimization of the filterability of crystals produced in such a cascade is dealt with, since a good filterability

determines, to a large extent, the velocity of crystallization and the uniformity of the conditions inside the vessel. In this paper several factors that affect boiling and circulation in vacuum pans are identified. The use of Computational Fluid Dynamics in the analysis of circulation and in the evaluation of alternatives for hydrodynamic optimization is discussed. Options for making changes to batch pans to improve the characteristics of the pan are identified, either to change the geometry or assist circulation. Some results with steam-assisted circulation (steam jiggers) are given, which show how the performance of a pan can be improved.

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In order to obtain constant solid properties with particles exhibiting a low order of symmetry, it is necessary to monitor and to control several distributed parameters characterizing the crystal shape and size. A bi-dimensional population balance model was

developed to simulate the time variations of two characteristic sizes of crystals. The nonlinear population balance equations were solved numerically over the bi-dimensional size domain using the so-called method of classes. An effort was made to improve usual simulation studies through the introduction of physical knowledge in the kinetic laws involved during nucleation and growth phenomena of complex organic products. The performances of the simulation algorithm were successfully assessed through the reproduction of two well-known theoretical and experimental features of ideal continuous crystallization processes: the computation of size-independent growth rates from the plot of the steady-state crystal size distribution and the possibility for MSMPR crystallizers to exhibit low-frequency oscillatory behaviors in the case of insufficient secondary nucleation.

Sima, M.A. and Harris, J.A. (1999)

In this paper one and three dimensional numerical models for the crystallization of sucrose from low grade massecuite are presented. Crystal growth is simulated by solving population balance equations for the moments of the crystal size distribution together with a transport equation describing sucrose mass balance. The models require the flow and temperature field to be known, and these are either specified for the one-dimensional model or obtained from solution of the mass, momentum and energy equations for the two dimensional model. The models are applied to study crystal growth in a vertical continuous cooling crystallizer. The simulations show that the vessel studied does not perform satisfactory as the predicted crystal growth is less than desired. The results illustrate some of the problems associated with cooling crystallization in highly viscous fluids, demonstrate the feasibility of simulating crystallization process using CFD, and display the enormous potential that CFD provides to improve crystallizer design.

In the cooling crystallizer studied the overall crystal growth was less than desirable, primarily due to a poor residence time distribution and a low mean shear rate. To improve the performance of this vessel the residence time distribution would have to be addressed (e.g. through the use of baffles to prevent the short circuiting) and, ideally, the mean shear rate increased so that mass transfer does not limit the growth rate.

Dixon, T., Hobson, P., Mann, A., Plaza, F., Pennisi, S. and Steindl, R. (2003)

This paper summarizes a wide range of ongoing applications of CFD in the sugar industry. Principal among these have been the development of bagasse combustion technologies and understanding the processes involved in bagasse combustion and steam generation. SRI has been active for over 10 years in CFD with the FURNACE code, encompassing the full range of applications from fundamental code development, the generation and commercialization of new ideas and technologies, to the resolution of practical plant problems. Typical areas have included particle erosion, corrosion and heat transfer in tube bundle units, particle drying, ignition and burnout dynamics, the development of advanced boiler designs and more recently CO and NO_x pollutant generation and reduction.

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Jansens, P.J., Bruinsma, O.S.L. and Rosmalen, G.M.V. (1995)

Cascades of crystallizers are applied in many processes. The optimization of the filterability of crystals produced in such a cascade is dealt with, since a good filterability

is often one of the main product specifications. An integral model description of the crystallization process is developed that allows for the prediction of the crystal size distribution and the filterability in each stage of the cascade, and experiments are performed to support this model. The integral model can be used as a predictive tool in future filterability optimization studies.

MATHEMATICAL MODELLING

The mathematical model proposed in this work to describe the steady state behavior of sugar crystallizer is based on mass and energy balance equations. It is set up on the following simplifying assumptions:

1. Crystal growth rate is assumed to be size independent. This is a reasonable assumption because size dependent phenomenon is not apparent in sugar crystallization as it is reported by many workers.
2. Impurities (non-sugars) are transferred from the solution to the crystals such that crystal purity remains constant during the operation.
3. Attrition, crystal breakage and agglomeration phenomena are generally neglected in crystallization processes This approximation is justified in the case of sugar crystallization because of the low shear involved and because of the fact that agglomeration is not usually significant for sucrose.
4. Because of the very small value of its flow rate, the superheated steam used for mixing the sugar suspension was neglected.

The balance equations, over compartment i, are described as follows:

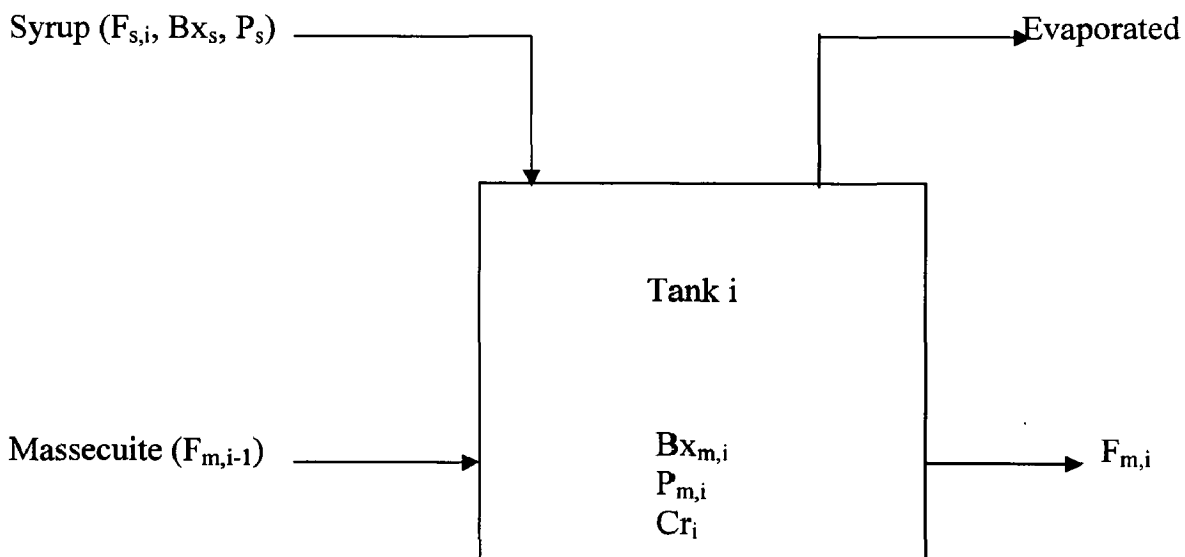


Fig. 3.1 Block Diagram of a Crystallizer Compartment

3.1 MATERIAL BALANCES

3.1.1 Water balance for the massecuite:

Input – Output + Generation – Accumulation = 0

$$\text{Input} = F_{m,i-1} \left(1 - \frac{Bx_{m,i-1}}{100} \right) + F_{s,i} \left(1 - \frac{Bx_s}{100} \right)$$

$$\text{Output} = F_{m,i} \left(1 - \frac{Bx_{m,i}}{100} \right) + F_{v,i}$$

Generation = 0

Accumulation = 0

Overall Balance

$$F_{m,i-1} \left(1 - \frac{Bx_{m,i-1}}{100} \right) + F_{s,i} \left(1 - \frac{Bx_s}{100} \right) - F_{m,i} \left(1 - \frac{Bx_{m,i}}{100} \right) + F_{v,i} + 0 - 0 = 0$$

$$F_{m,i-1} \left(1 - \frac{Bx_{m,i-1}}{100} \right) + F_{s,i} \left(1 - \frac{Bx_s}{100} \right) - F_{m,i} \left(1 - \frac{Bx_{m,i}}{100} \right) - F_{v,i} = 0 \quad (3.1)$$

3.1.2 Sucrose balance for the solution:

Input – Output + Generation – Accumulation = 0

$$\text{Input} = F_{m,i-1} \left(\frac{Bx_{m,i-1} P_{m,i-1}}{10000} - \frac{Cr_{i-1} Pc}{10000} \right) + F_{s,i} \left(\frac{Bx_s P_s}{10000} \right)$$

$$\text{Output} = F_{m,i} \left(\frac{Bx_{m,i} P_{m,i}}{10000} - \frac{Cr_i Pc}{10000} \right)$$

Generation = 0

Accumulation = $Scr_i r_{c,i}$

Overall Balance

$$F_{m,i-1} \left(\frac{Bx_{m,i-1} P_{m,i-1}}{10000} - \frac{Cr_{i-1} Pc}{10000} \right) + F_{s,i} \left(\frac{Bx_s P_s}{10000} \right) - F_{m,i} \left(\frac{Bx_{m,i} P_{m,i}}{10000} - \frac{Cr_i Pc}{10000} \right) + 0 -$$

$$Scr_i r_{c,i} = 0$$

$$F_{m,i-1} \left(\frac{Bx_{m,i-1} P_{m,i-1}}{10000} - \frac{Cr_{i-1} Pc}{10000} \right) + F_{s,i} \left(\frac{Bx_s P_s}{10000} \right) - F_{m,i} \left(\frac{Bx_{m,i} P_{m,i}}{10000} - \frac{Cr_i Pc}{10000} \right) - Scr_i r_{c,i} = 0 \quad (3.2)$$

3.1.3 Non-sugars Balance for the massecuite:

Input – Output + Generation – Accumulation = 0

$$\text{Input} = F_{m,i-1} \frac{Bx_{m,i-1}}{100} \left(1 - \frac{P_{m,i-1}}{100}\right) + F_{s,i} \frac{Bx_s}{100} \left(1 - \frac{P_s}{100}\right)$$

$$\text{Output} = F_{m,i} \frac{Bx_{m,i}}{100} \left(1 - \frac{P_{m,i}}{100}\right)$$

Generation = 0

Accumulation = 0

Overall Balance

$$F_{m,i-1} \frac{Bx_{m,i-1}}{100} \left(1 - \frac{P_{m,i-1}}{100}\right) + F_{s,i} \frac{Bx_s}{100} \left(1 - \frac{P_s}{100}\right) - F_{m,i} \frac{Bx_{m,i}}{100} \left(1 - \frac{P_{m,i}}{100}\right) + 0 - 0 = 0$$

$$F_{m,i-1} \frac{Bx_{m,i-1}}{100} \left(1 - \frac{P_{m,i-1}}{100}\right) + F_{s,i} \frac{Bx_s}{100} \left(1 - \frac{P_s}{100}\right) - F_{m,i} \frac{Bx_{m,i}}{100} \left(1 - \frac{P_{m,i}}{100}\right) = 0 \quad (3.3)$$

3.1.4 Crystallized sugar balance for the massecuite:

Input – Output + Generation – Accumulation = 0

$$\text{Input} = F_{m,i-1} \frac{Cr_{i-1}Pc}{10000}$$

$$\text{Output} = F_{m,i} \frac{Cr_iPc}{10000}$$

Generation = $Scr_i r_{c,i}$

Accumulation = 0

Overall Balance

$$F_{m,i-1} \frac{Cr_{i-1}Pc}{10000} - F_{m,i} \frac{Cr_iPc}{10000} + Scr_i r_{c,i} - 0 = 0$$

$$F_{m,i-1} \frac{Cr_{i-1}Pc}{10000} - F_{m,i} \frac{Cr_iPc}{10000} + Scr_i r_{c,i} = 0 \quad (3.4)$$

3.2 HEAT BALANCE

Input – Output + Generation – Accumulation = 0

$$\text{Input} = F_{m,i-1}h_{m,i-1} + F_{s,i}h_{s,i}$$

$$\text{Output} = F_{m,i}h_{m,i} + F_{v,i}h_{v,i}$$

$$\text{Generation} = U_i Sc (T_{vc} - T_m)$$

$$\text{Accumulation} = Scr_i r_{c,i} h_{c,i}$$

Overall Balance

$$F_{m,i-1}h_{m,i-1} + F_{s,i}h_{s,i} - F_{m,i}h_{m,i} + F_{v,i}h_{v,i} + U_i Sc (T_{vc} - T_m) - Scr_i r_{c,i} h_{c,i} = 0 \quad (3.5)$$

In Eqs. (3.1)–(3.5), $Bx_{m,i}$, $F_{m,i}$ and $P_{m,i}$ represent the brix, the mass flow rate and the purity of the massecuite, respectively; Bx_s , $F_{s,i}$ and P_s are the same characteristics of liquor/syrup; Cr_i and P_c , the crystal content and purity (=98%), respectively; $h_{m,i}$ and h_s , the specific enthalpy of massecuite and syrup, respectively; $F_{v,i}$, the mass flow rate of evaporated water; $h_{v,i}$, the latent heat of water vaporization; U_i and Sc , the heat transfer coefficient and surface area, respectively; T_m , T_{vc} , the massecuite and the heating steam temperatures, respectively; $h_{c,i}$, the enthalpy of crystallization; Scr_i , the total surface area of crystals; $r_{c,i}$ is the crystal growth rate, and $G_{c,i}$ is the corresponding linear rate.

3.3 PHASE EQUILIBRIUM

The phase equilibrium property considered is the over saturation (OS). Also considered is the nucleation limit defined by the critical over saturation ($OScrit$). The following section defines and develops the over saturation and critical over saturation relationships.

The solubility of sucrose is defined as the concentration of sucrose in a saturated solution, which is in equilibrium with sucrose in the solid state. The solubility of a pure sucrose solution is temperature dependent and the following equation was given by Charles (1960).

$$\left(\frac{S}{S+W}\right)^* = \frac{\left(64.407 + 7.251\left(\frac{T}{100}\right) + 20.57\left(\frac{T}{100}\right)^2 - 9.04\left(\frac{T}{100}\right)^3\right)}{100}$$

When linearized to 65⁰C the equation becomes,

$$\left(\frac{S}{S+W}\right)^* = 0.7533 + 0.00225(T - 65) \quad (3.6)$$

This equation can now be converted to the mass ratio of sucrose to water which varies with temperature is

$$\left(\frac{S}{W}\right)^* = \frac{0.7533 + 0.00225(T - 65)}{1 - (0.7533 + 0.00225(T - 65))} \quad (3.7)$$

Supersaturation is defined as,

$$SS = \frac{\left(\frac{S}{W}\right)}{\left(\frac{S}{W}\right)^*} \quad (3.8)$$

If the solution is saturated then the value of SS will be 1, while if it is over-saturated the value is greater than 1 and if under-saturated the value is less than 1.

In practice sucrose solutions contain impurities and hence SS is redefined as;

$$SS = \frac{\left(\frac{S}{W}\right)}{Sc\left(\frac{S}{W}\right)^*} \quad (3.9)$$

Where SC (saturation coefficient) is the ratio of the impure sucrose equilibrium concentration to that of the pure sucrose equilibrium concentration.

Various authors have given correlations for the saturation coefficient. Some of those are,

- Wright (1971) suggested

$$SC = 1 - \left(\frac{I}{W}\right) \quad (3.10)$$

- Wright and White (1968) gave another relationship, which was temperature dependent;

$$SC = 1 - \left(0.1 - \frac{T - 60}{200}\right) \left(\frac{I}{W}\right) \quad (3.11)$$

- Broadfoot and Steindl (1980) later presented a correlation for Queensland conditions

$$SC = 1 - \frac{5.75}{T} \left(\frac{I}{W} \right)^{(0.1+0.28RS/ash)} \quad (3.12)$$

A more recent correlation presented by Schneider (1996) that was proposed by SRI (Sugar Research Institute) is;

$$Sc = P_5 \left(\frac{I}{W} \right) + P_6 + (1 - P_6) \exp \left(-P_7 \frac{I}{W} \right) \quad (3.13)$$

where

$$P_5 = 0.011 + 0.00046T$$

$$P_6 = 0.67 + 0.0021T - 0.07RS / ash$$

$$P_7 = 0.54 + 0.0049T$$

Over saturation is defined as

$$OS = SS - 1 \quad (3.14)$$

An equation for the critical over saturation is given by White (2000) as,

$$OS_{crit} = 0.11 + 3.6 \left(\frac{I}{S + I} \right)^3 \quad (3.15)$$

The driving force for crystallization is over saturation, which is a measure of the sucrose content above equilibrium in the molasses. As shown in equation when the impurities increase, the critical over saturation increases. One of the major objectives with efficient pan boiling is to avoid nucleation in the pan so the over saturation must be controlled so that it remains below the limits of the critical over saturation i.e. below the secondary nucleation limit.

3.4 CRYSTAL GROWTH RATE RELATIONS

The growth of crystals is defined in terms of the normalized first moment as follows;

$$G = \frac{du_1}{dt} \quad (3.16)$$

Several different experimental growth rate expressions have been used in past work. Wright (1971) considered the effect of temperature and impurities on over saturation. The over saturation gives a measure of the excess sucrose to water in the pan

at any time. The effect of OS/G is non-linear. As shown in *equation 24* the results may be fitted by a displaced linear relationship for high over saturation while at low over saturation (*equation 25*) the linear relationship may be used. Further *equation 26* shows when at negative over saturations dissolution occurs.

$$OS_{crit} > OS > 1.5P_2 \quad G = K_T(OS - P_2) \quad (3.17)$$

$$1.5P_2 > OS > 0 \quad G = \frac{K_T}{3}OS \quad (3.18)$$

$$0 > OS \quad G = 5K_T OS \quad (3.19)$$

P_2 is the over saturation to change from the low over saturation (OS) equation to the higher over saturation. This value of k_T applies at a given temperature and impurity level.

The effect of temperature is correlated by an Arrhenius relation with variable activation energy,

$$K_T = K_{60^\circ} e^{\left[\frac{E_{act}}{R} \left(\frac{1}{T+273.16} - \frac{1}{333.16} \right) \right]} \quad (3.20)$$

where

$$E_{act} = 62.76 - 0.8368(T - 60) \quad (3.21)$$

The K_{60} term includes the effect of impurities, which is taken as varying exponentially with the impurities to water ratio.

$$K_{60^\circ} = P_1 e^{\left(P_3 \frac{I}{W} \right)} \quad (3.22)$$

Wright (1971) estimated the three growth related parameters to be;

$$P_1 = 7.418 \quad \left[\frac{mm}{h.OS} \right]$$

$$P_2 = 0.04 \quad [OS]$$

$$P_3 = -1.75 \quad \text{unitless}$$

Therefore the relation for growth rate as given by Wright (1971) is;

$$G = P_1 e^{\left(\frac{P_3 I}{W}\right)} e^{\left(\frac{-E_{act}}{R} \left(\frac{1}{T+273.16} - \frac{1}{333.16}\right)\right)} (OS - P_2) \left[\frac{mm}{h} \right] \text{ for } OS > 1.5 P_2$$

$$G = P_1 e^{\left(\frac{P_3 I}{W}\right)} e^{\left(\frac{-E_{act}}{R} \left(\frac{1}{T+273.16} - \frac{1}{333.16}\right)\right)} \frac{K_T}{3} OS \left[\frac{mm}{h} \right] \text{ for } 1.5 P_2 > OS > 0$$

$$G = P_1 e^{\left(\frac{P_3 I}{W}\right)} e^{\left(\frac{-E_{act}}{R} \left(\frac{1}{T+273.16} - \frac{1}{333.16}\right)\right)} 5K_T OS \left[\frac{mm}{h} \right] \text{ for } OS < 0$$

Therefore the relation for crystal growth rate is given by

$$r_i = 6G_i \frac{K_v P_c}{K_a} \quad (3.23)$$

where

K_v = volume shape factor

K_a = surface shape factor

3.5 ESTIMATION OF MODEL PARAMETERS

Estimation of the heat transfer coefficient, U is performed using empirical correlation proposed for evaporating crystallizers and evaporators in sugar industry.

$$U_i = 2136.4 \frac{T_m}{Bx_{m,i}} \quad (3.24)$$

where

T_m = Temperature of the massecuite

$Bx_{m,i}$ = Brix of the massecuite

With the assumption that the numbers of crystals remain constant all along the strike, the total surface area can be expressed according to the weight of the crystals.

$$SCr_i = \frac{K_a N^{1/3}}{(PK_v)^{2/3}} C^{2/3} \quad (3.25)$$

Where K_a is the surface shape factor of a crystal, K_v is the volume shape factor, P is the density of sucrose crystals, N is the number of crystals and C is the mass of crystals

in the pan. The values of these parameters are supposed to be known and taken from literature.

Moments of the crystal size distribution:

$$\frac{d(u_j)}{dt} - j \int_0^{\infty} L^{j-1} G(L,t) f(L,t) dL = 0 \quad (3.26)$$

If G is independent of L (Mac cabe's law)

$$\frac{d(u_j)}{dt} - jGu_{j-1} = 0$$

According to the assumptions (no nucleation or no growth rate dispersion)

$$\frac{d(u_0)}{dt} = 0$$

$$\frac{d(u_1)}{dt} = G$$

$$\frac{d(u_2)}{dt} = 2Gu_1$$

$$\frac{d(u_3)}{dt} = 3Gu_2$$

Where u_0 , u_1 (m), u_2 (m²), u_3 (m³) are moments of the crystal size distribution.

Now the crystals mean aperture can be calculated by using the moments of crystal size distribution.

$$MA = \frac{u_4}{u_3} \quad (3.27)$$

Where MA is the crystals mean aperture.

Enthalpy of massecuite and syrup can be calculated by using these correlations.

Enthalpy of massecuite can be calculated by

$$h_m = (-0.0324x^2 + 4.6027x - 167.44) + (-0.022x + 4.1084)T_m \quad (3.28)$$

where

h_m = enthalpy of massecuite

T_m = temperature of massecuite

x = concentration of massecuite

Enthalpy of syrup can be calculated by

$$h_s = (-0.001x^3 + 0.1744x^2 - 9.9213x + 177.11) + (-0.0241x + 4.2606)T_s \quad (3.29)$$

where

h_s = enthalpy of syrup

T_s = temperature of syrup

x = concentration of syrup

These correlations are developed using graph values for enthalpies of massecuite and for enthalpies of syrup. This has been found that values of enthalpies lie within the error range of 5%.

COMPUTATIONAL TECHNIQUE

This chapter presents a method for solution of the model equations which have been discussed in the preceding chapter, for a vacuum crystallizer of the sugar industry.

The material balances provide a set of equations, which have to be solved with the enthalpy balances to get an improved set of variables. The set of equations were solved by iterative method. Matlab program for the solution of model is given in appendix E. In this section an algorithm has been developed for the solution of balance equations.

4.1 ALGORITHM FOR CRYSTALLIZER CALCULATIONS

1. Calculate the value of crystal growth rate by using the equations given in chapter 3.
2. Assume the flow rate of massecuite for the calculation of crystal content.
3. Calculate the crystal content for assumed flow rate of massecuite by using the equation (3.4).
4. Calculate the values of flow rate of syrup and purity of massecuite with the help of equation (3.2) and equation (3.3).
5. Calculate the value of flow rate of evaporated water by using the equation (3.1).
6. Calculate the value of heat transfer coefficient with the help of equation (3.24).
7. Calculate the enthalpies of syrup and massecuite using the correlation given in appendix A.
8. Put the values of flow rate of massecuite, crystal content, purity of massecuite, flow rate of syrup and flow rate of evaporated water in heat balance equation, if the heat balance equation is not satisfied with these values, go to step 2 and assume new value for flow rate of massecuite else go to step 1 for the next iteration with the new values of crystal content, purity of massecuite and flow rate of massecuite.
9. Repeat the above procedure upto nine iterations.

RESULTS AND DISCUSSIONS

In this chapter an analysis of the results obtained by solving the model developed in chapter 3 by the technique as explained in chapter 4 is being done. In the following sections the model validity and the parametric sensitivity of the process is analyzed, using this model and the solution technique. The parameters considered for this study are crystal content, crystal mean aperture, purity of massecuite and brix of massecuite.

Data characterizing the actual operating conditions of the crystallizer employed are given in appendix A. These data were measured using on-line sensors and analysis techniques elaborated by the plant control laboratory. Massecuite samples were taken from different compartments using sampling devices available on the apparatus as well as a vacuum pump. Theoretical results of simulation by Bounahmidi, T. *et al* (2001) are given in appendix B and simulation results obtained by solving the model developed in chapter 3 by the technique as explained in chapter 4 are given in appendix C.

5.1 VALIDITY OF THE MODEL

Appendix C summarizes the main simulation results concerning the performance of the crystallizer. Appendix D gives the absolute errors observed between the measured values and these results.

For a better illustration of these results, measured values, theoretical values and those provided by the model are plotted for some of the operating variables in Fig. 5.1 and Fig. 5.2. Examination of these figures shows that the profiles provided by the model have practically the same shape as those industrially and theoretically observed.

Fig. 5.1 representing the evolution of the crystal content as a function of compartment number indicates a decrease of this variable in the first compartment, which is due to the abnormal conditions in this compartment, where the dissolution process takes place instead of the crystal growth. After first compartment crystal content increases with the compartments, which is due to the crystal growth rate is increasing with the compartments. Fig. 5.1 also shows a comparison between the crystal content

obtained with that due to Bounahmidi, T. *et al* (2001) and with the experiment values. There have been fairly good agreement with maximum deviation around 5 percent. The reasons for this may be error in the approximation of enthalpy data and error in the approximation of crystal growth rate.

Fig. 5.2 representing the evolution of the crystals mean aperture a function of compartment number indicates a decreases of this variable in the first compartment, which is due to the abnormal conditions in this compartment, where the dissolution process takes place instead of the crystal growth. After first compartment crystals mean aperture increases with the compartments. Fig. 5.2 also shows a comparison between the crystals mean aperture obtained with that due to Bounahmidi, T. *et al* (2001) and with the experiment values. There have been fairly good agreement with maximum deviation around 5 percent. The reasons for this may be error in the approximation of enthalpy data and error in the approximation of crystal growth rate.

It can be showed from the above discussion that the results obtained from the developed model for the crystallizer, are in moderately good agreement with the real data measured on the industrial installation and with the theoretical results and allow adequate estimation of the main observed trends. Therefore, the simulation model is of a satisfactory accuracy and can be used for the monitoring of Industrial continuous crystallizer of first grade massequite as that employed in the current work.

5.2 PARAMETRIC SENSITIVITY STUDY

In this section an effect of operating variables for a crystallization process, which for this process are the crystal content, crystals mean aperture, purity of massequite and brix of massequite has been studied. The variables on which the analysis has been done have been varied in the range as given below

Crystal contents	10-52 (wt. %)
Crystals mean aperture	179-275 m
Purity of massequite	89.08-89.22 (wt. %)
Brix of massequite	79-93 (wt. %)

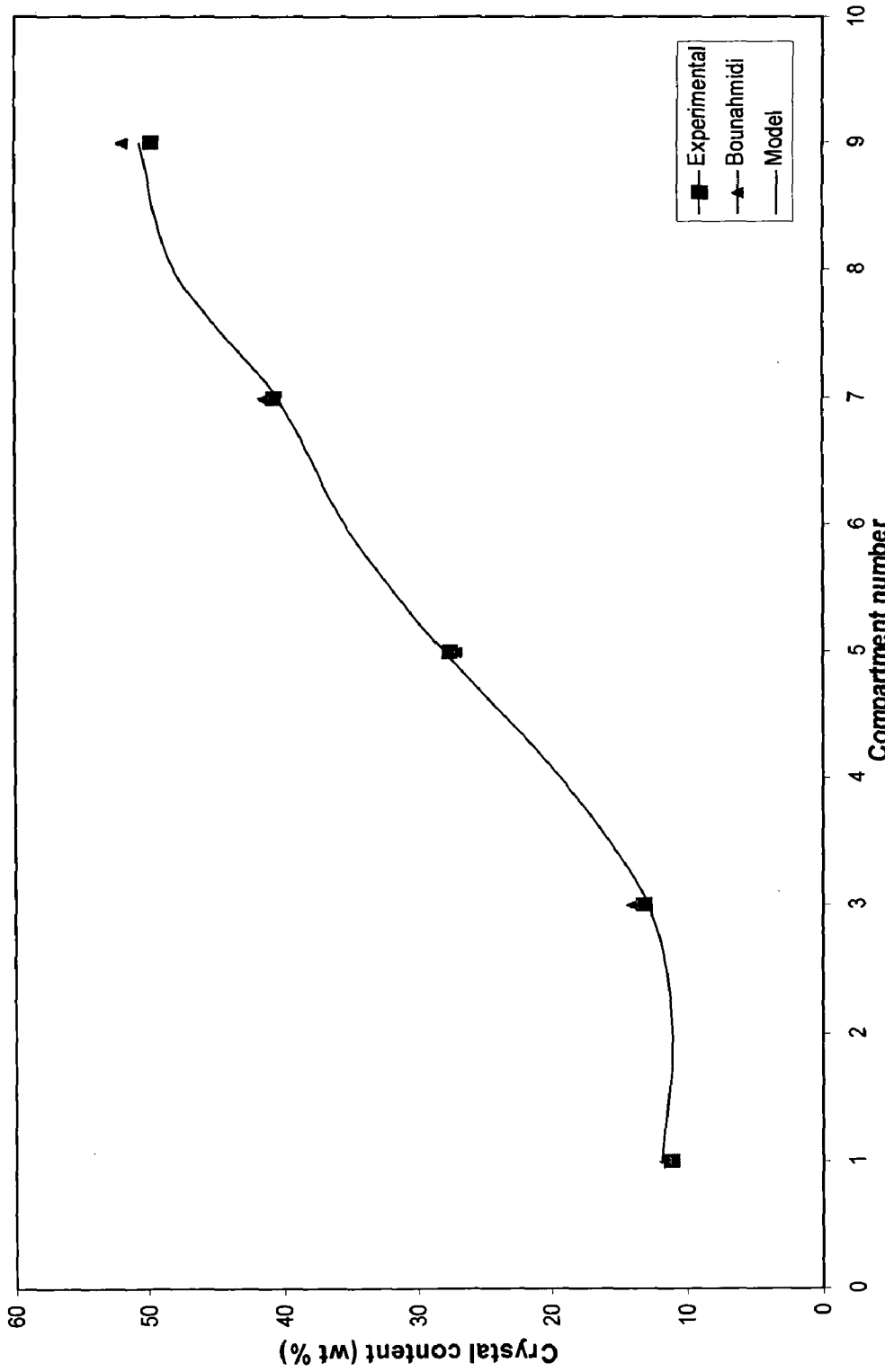


Fig. 5.1 Evolution of crystal content along the crystallizer

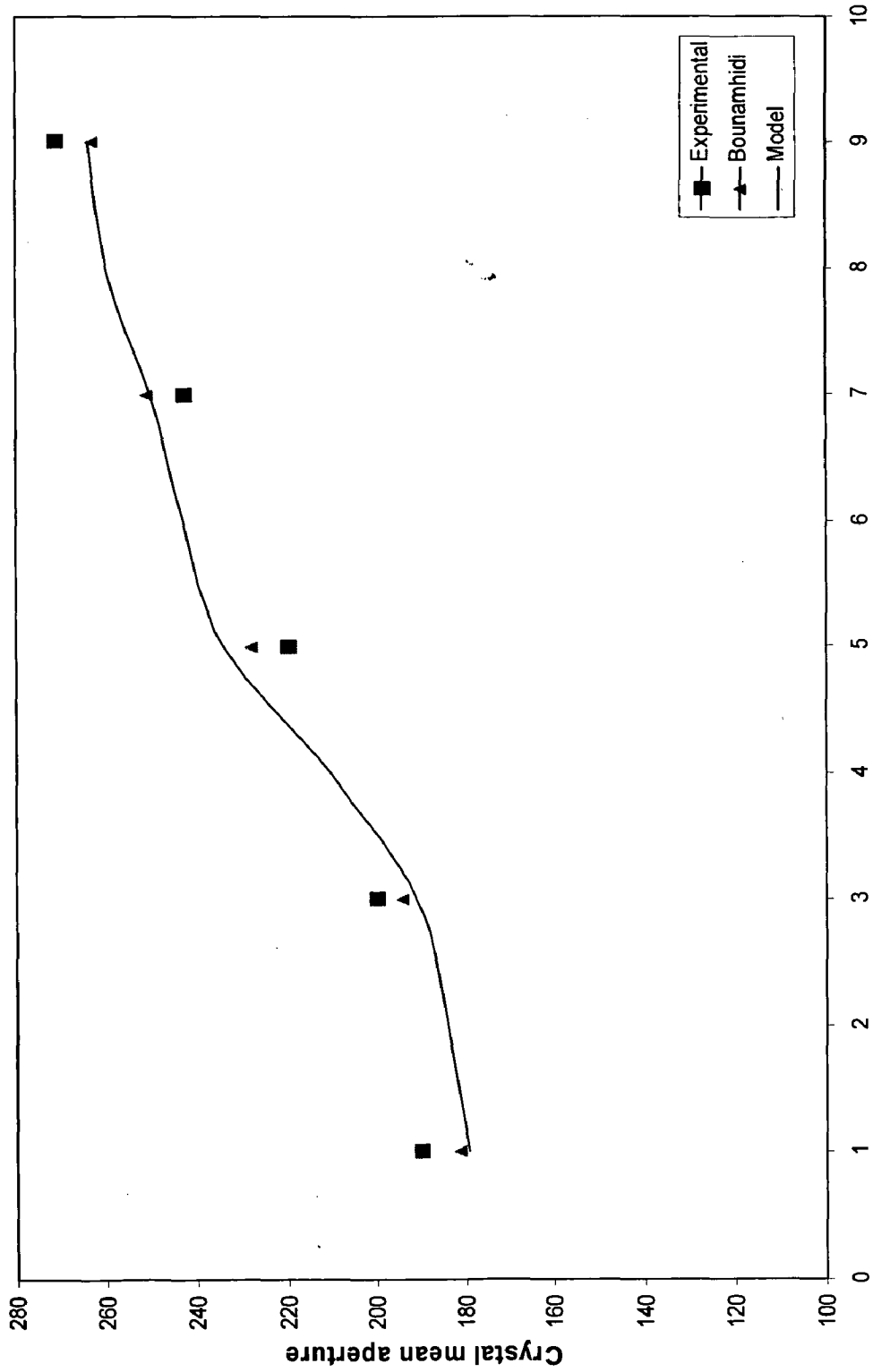


Fig. 5.2 Evolution of crystal mean size along the crystallizer

5.2.1 EFFECT OF CRYSTAL CONTENT

The effect of crystal content which is fed to compartments on the brix of massecuite, flow rates of massecuite, syrup and evaporated water, on purity of massecuite and on crystals mean aperture is illustrated from fig. 5.3 to fig. 5.8.

Fig. 5.3 shows the effect of crystal content on the brix of massecuite. As the crystal content increases brix of massecuite also increases, because with the process water will be evaporated so quantity of water will be decreases and brix of massecuite increases.

Fig. 5.4 shows the effect of crystal content on the flow rate of massecuite. In starting flow rate of massecuite increases with increases in crystal content but after that flow rate of massecuite decreases with increases in crystal content, because as the crystal content increases that means number of crystals per unit area also increases so due to this effect flow rate of massecuite decreases.

Fig. 5.5 shows the effect of crystal content on the crystals mean aperture. It shows that crystal mean aperture increases with increase in the crystal content. In the starting as crystal content decreases crystals mean aperture also decreases.

Fig. 5.6 shows the effect of crystal content on the flow rate of evaporated water. It shows that flow rate of evaporated water decreases with increase in the crystal content, because as the crystal content increases the brix of massecuite also increases and water decreases, it also shown in fig. 5.3, so due to this effect flow rate of evaporated water also decreases.

Fig. 5.7 shows the effect of crystal content on the flow rate of syrup. It shows that flow rate of syrup decrease with increase in the crystal content, because as the crystal content increases the brix of massecuite also increases so we needed less syrup for the process.

Fig. 5.8 shows the effect of crystal content on the purity of massecuite. It shows that purity of massecuite continuously increases with increase in the crystal content. In the starting purity of massecuite increases very rapidly after that it increases very slowly.

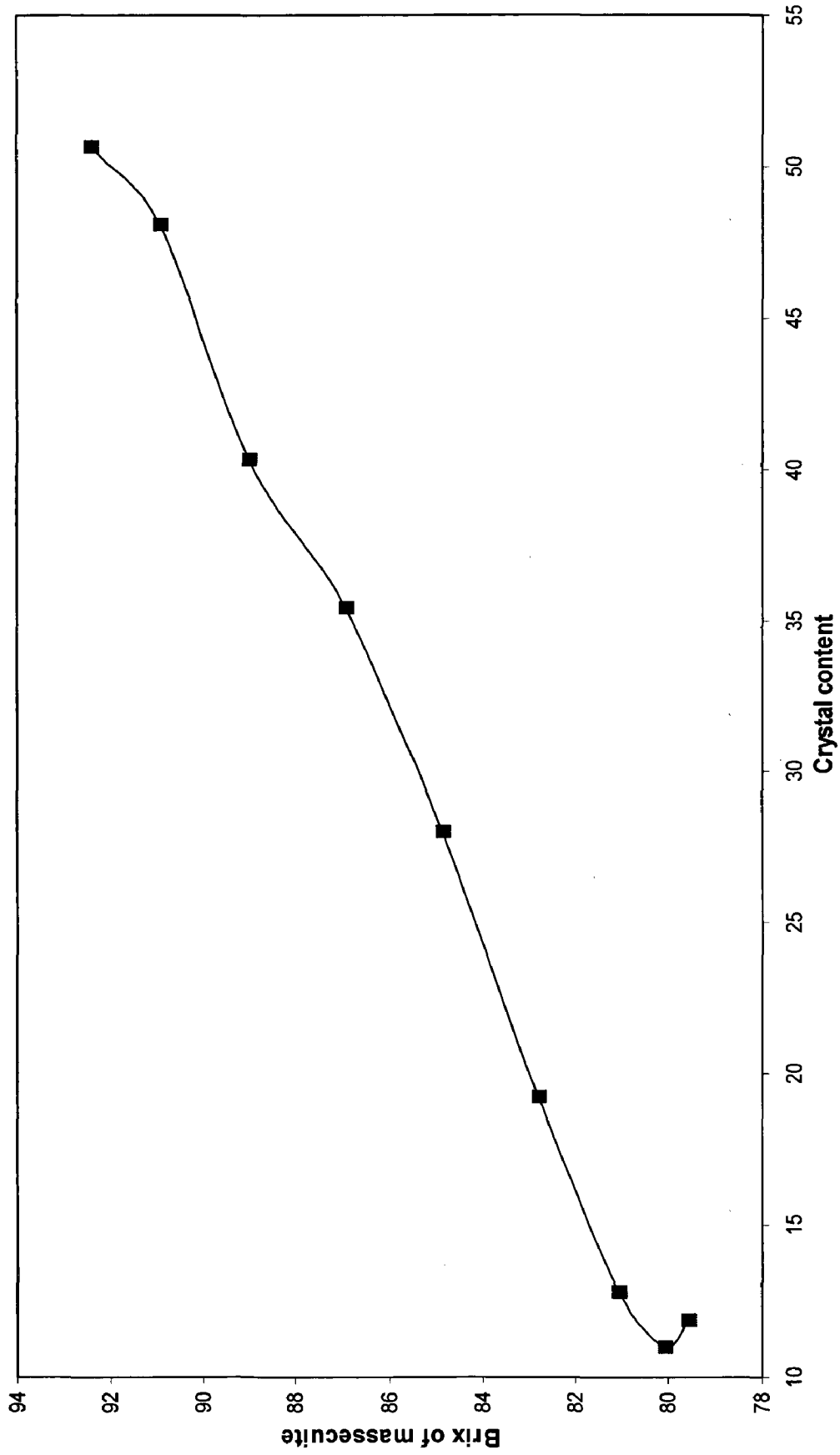


Fig. 5.3 Effect of crystal content on brix of massecuite

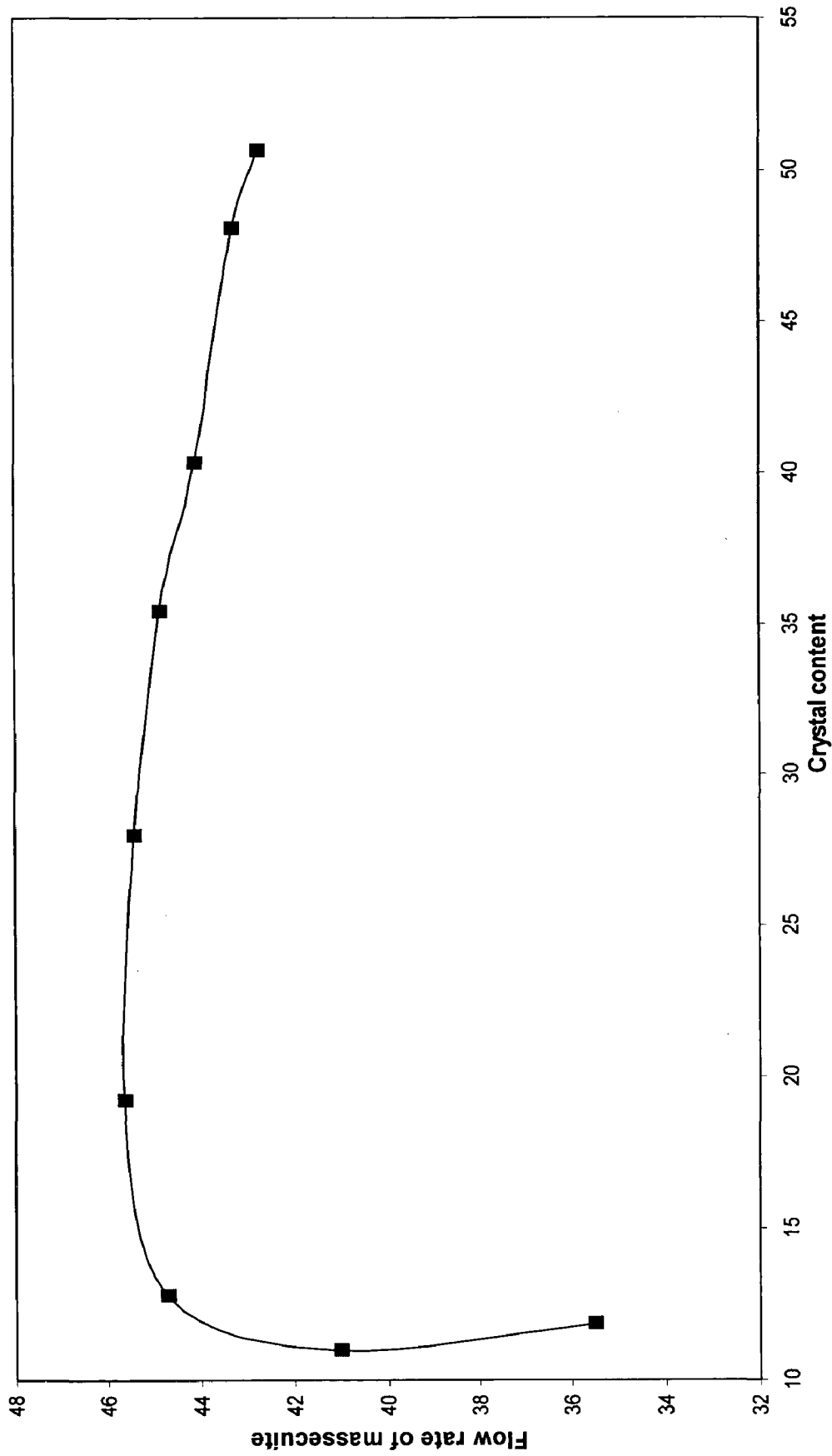


Fig. 5.4 Effect of crystal content on flow rate of massescite

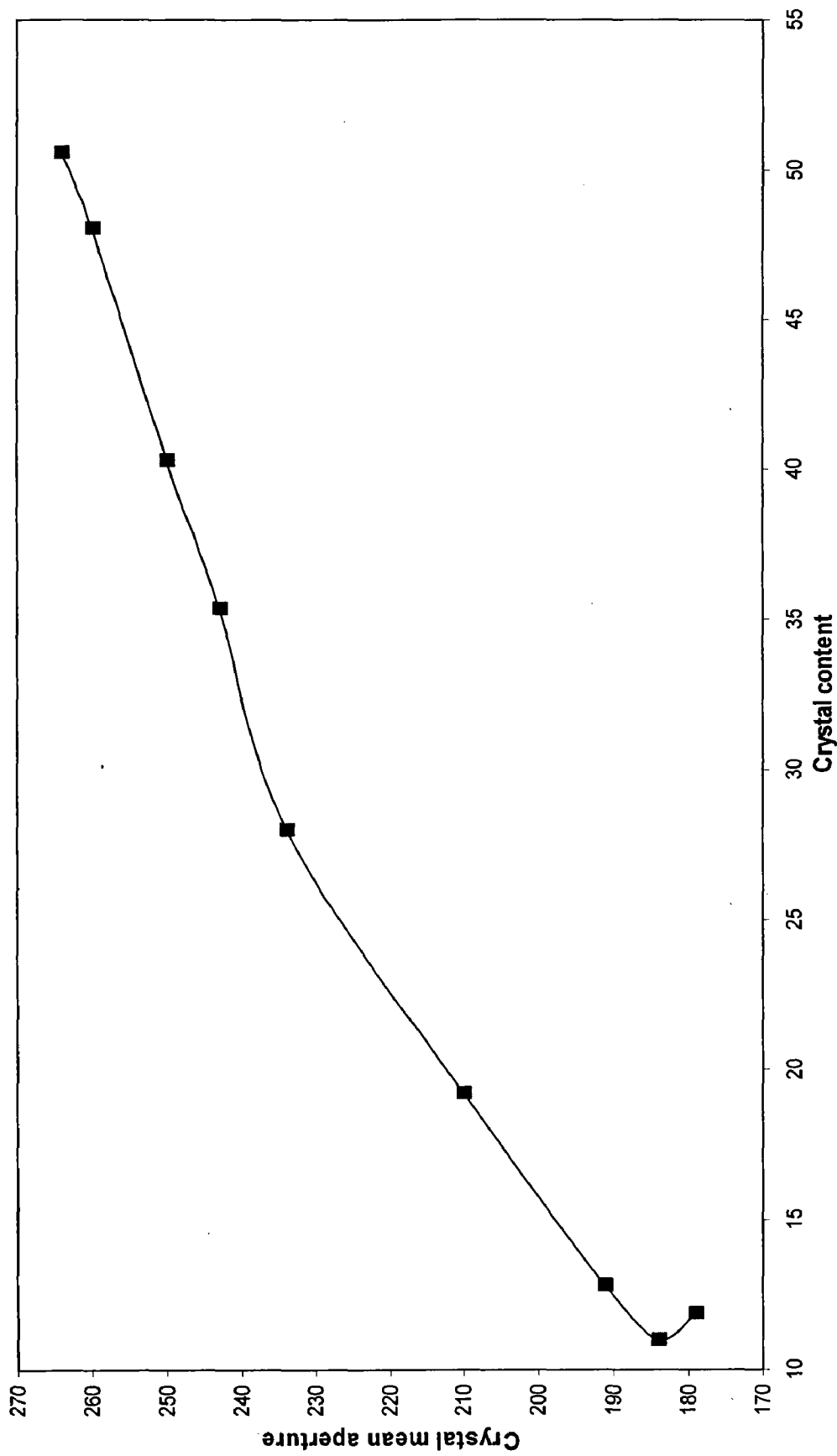


Fig. 5.5 Effect of crystal content on crystal mean aperture

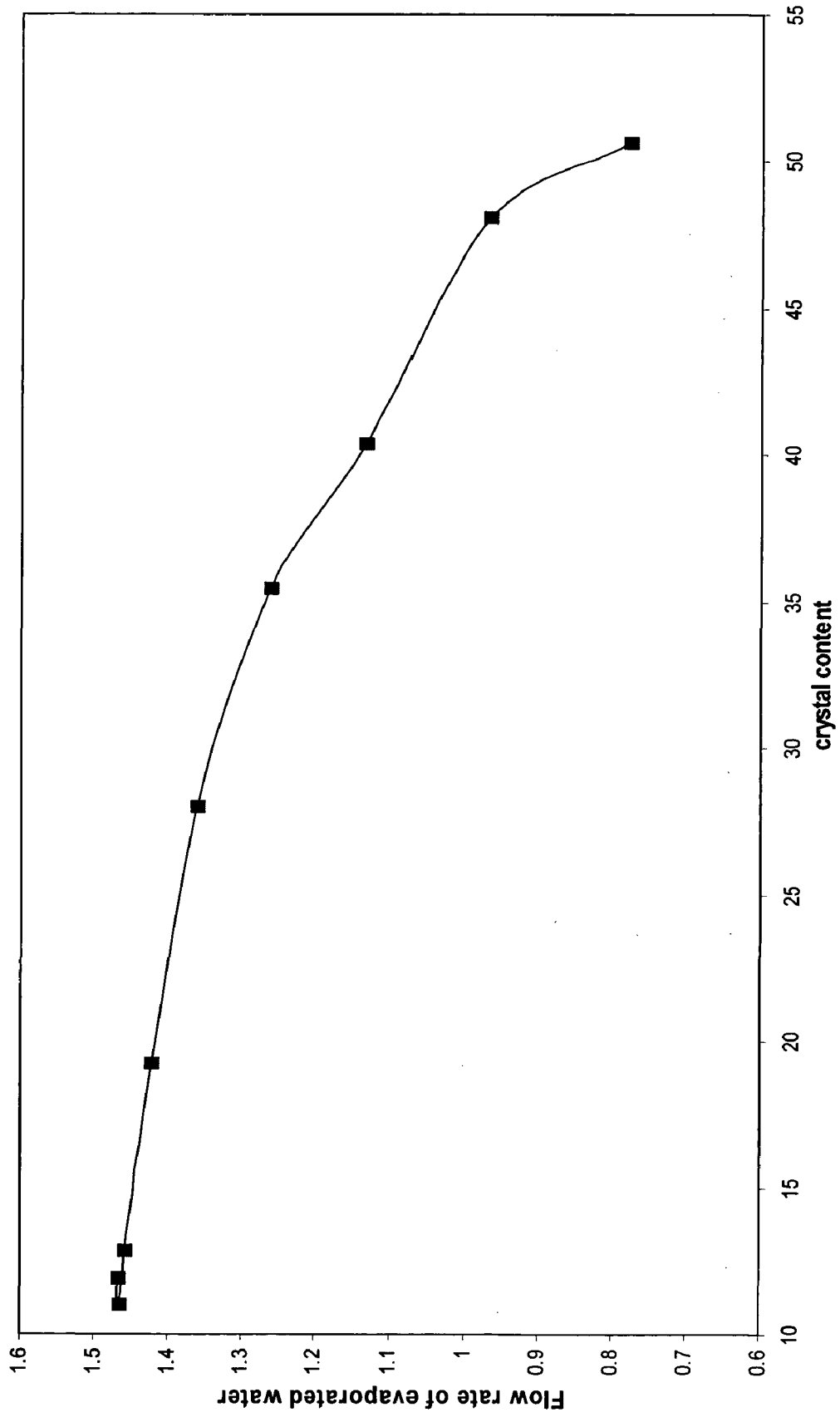


Fig. 5.6 Effect of crystal content on flow rate of evaporated water

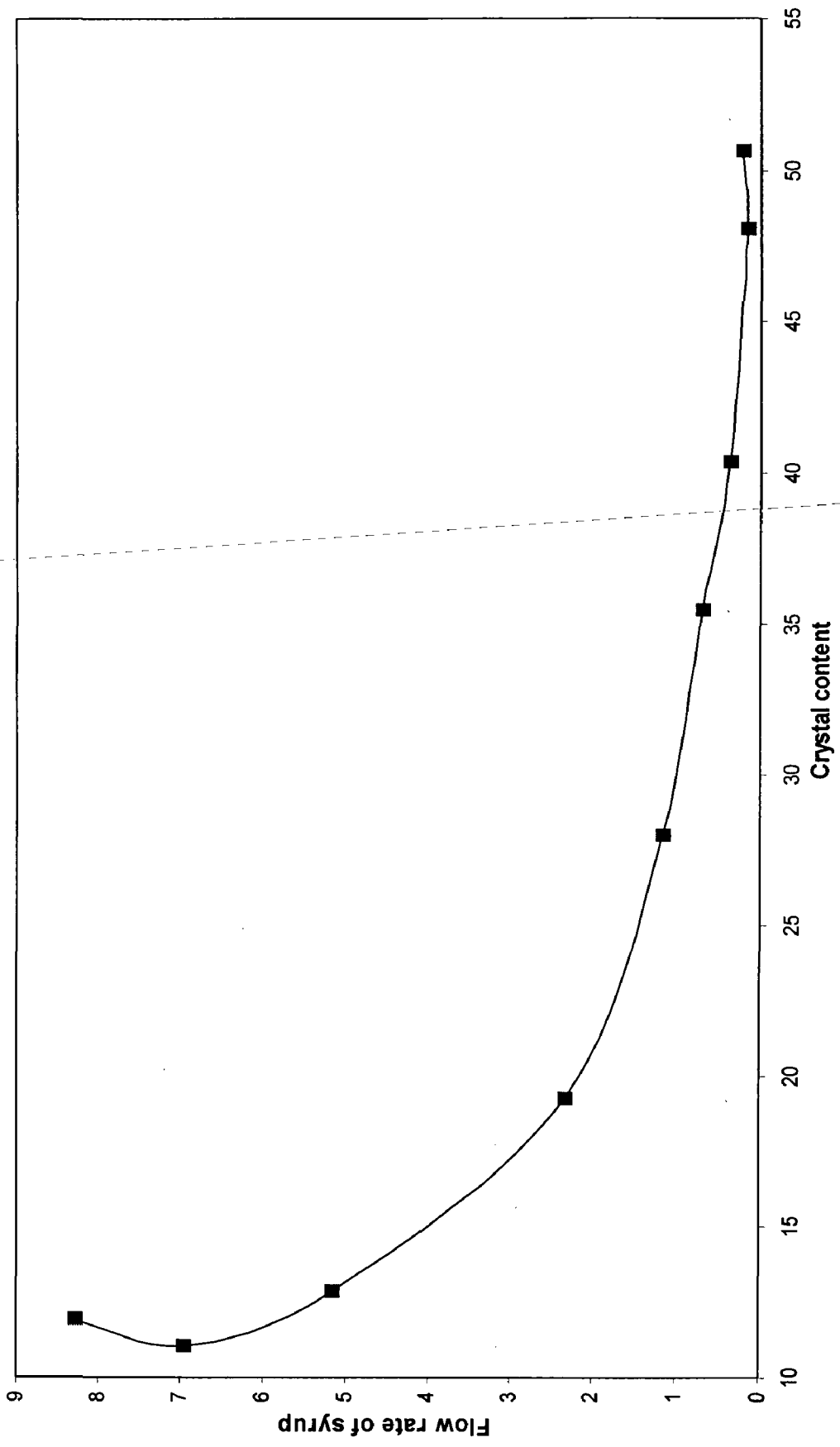


Fig. 5.7 Effect of crystal content on flow rate of syrup

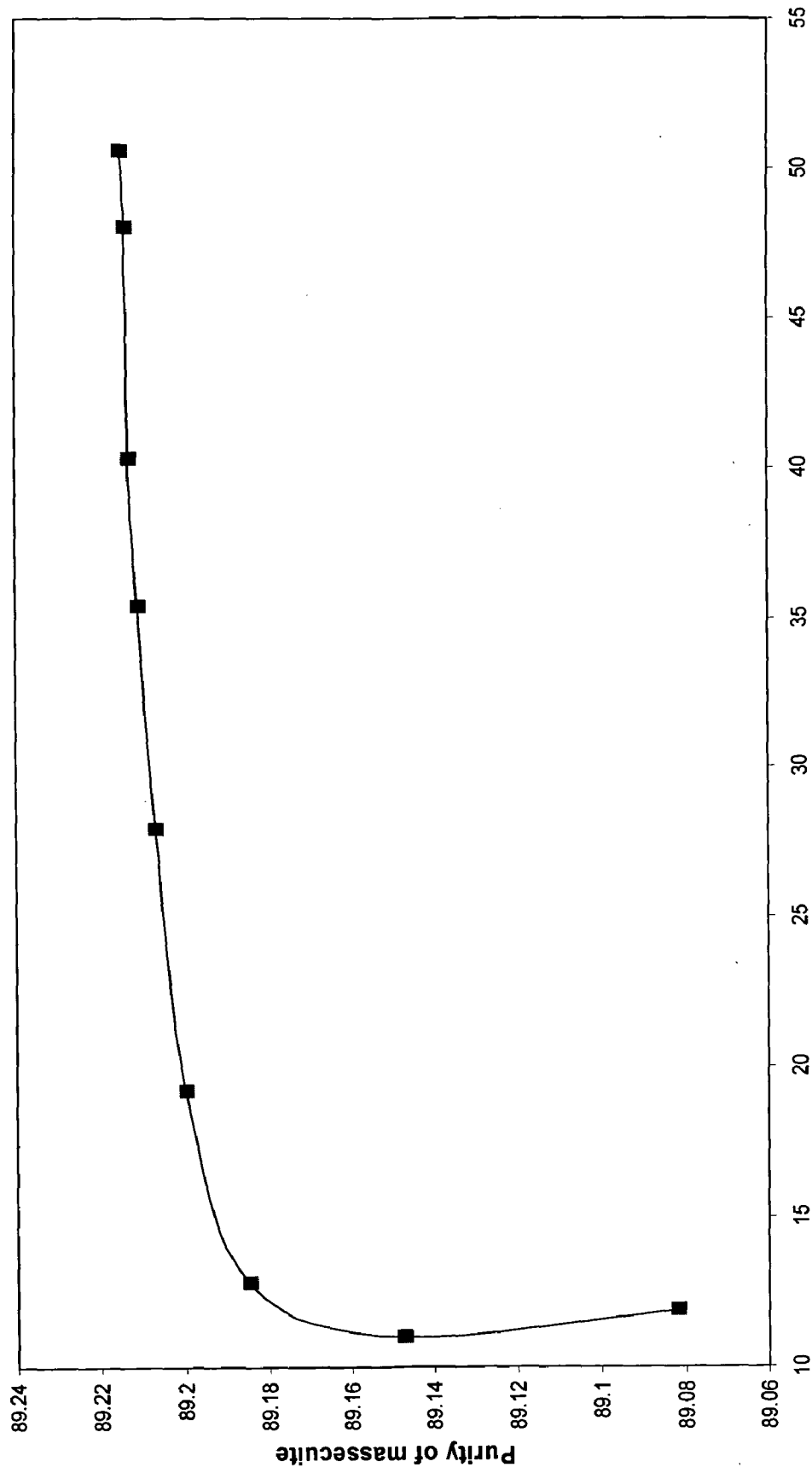


Fig. 5.8 Effect of crystal content on purity of masseccite

5.2.2 EFFECT OF CRYSTALS MEAN APERTURE

The effect of crystals mean aperture on the brix of massecuite, flow rates of massecuite, syrup and evaporated water and on purity of massecuite illustrated from fig. 5.9 to fig. 5.13.

Fig. 5.9 shows the effect of crystals mean aperture on the brix of massecuite. It shows that brix of massecuite increases with increase in the crystals mean aperture, because as the crystals mean aperture increases crystal content also increases, it also shown in fig. 5.3, as the crystal content increases brix of massecuite increases, so due to this effect brix of massecuite increases with increase in the crystals mean aperture.

Fig. 5.10 shows the effect of crystals mean aperture on the flow rate of massecuite. It shows that in starting flow rate of massecuite increases with increase in crystals mean aperture after that flow rate of massecuite decreases with increase in the crystals mean aperture, because as the crystals mean aperture increases crystal content also increases.

Fig. 5.11 shows the effect of crystals mean aperture on the flow rate of evaporated water. It shows that flow rate of evaporated water decreases with increase in the crystals mean aperture, because as the crystals mean aperture increases the crystal content increases, as the crystal content increase the brix of massecuite also increases and water decreases, it also shown in fig. 5.3, so due to this effect flow rate of evaporated water also decreases.

Fig. 5.12 shows the effect of crystals mean aperture on the flow rate syrup. It shows that flow rate of syrup decreases with increase in the crystals mean aperture, because with increases in the crystals mean aperture, crystal contents also increases and as the crystal content increases the brix of massecuite also increases so we needed less syrup for the process.

Fig. 5.13 shows the effect of crystals mean aperture on the purity of massecuite. It shows that purity of massecuite continuously increases with increase in the crystals mean aperture. In the starting purity of massecuite increases very rapidly after that it increases very slowly.

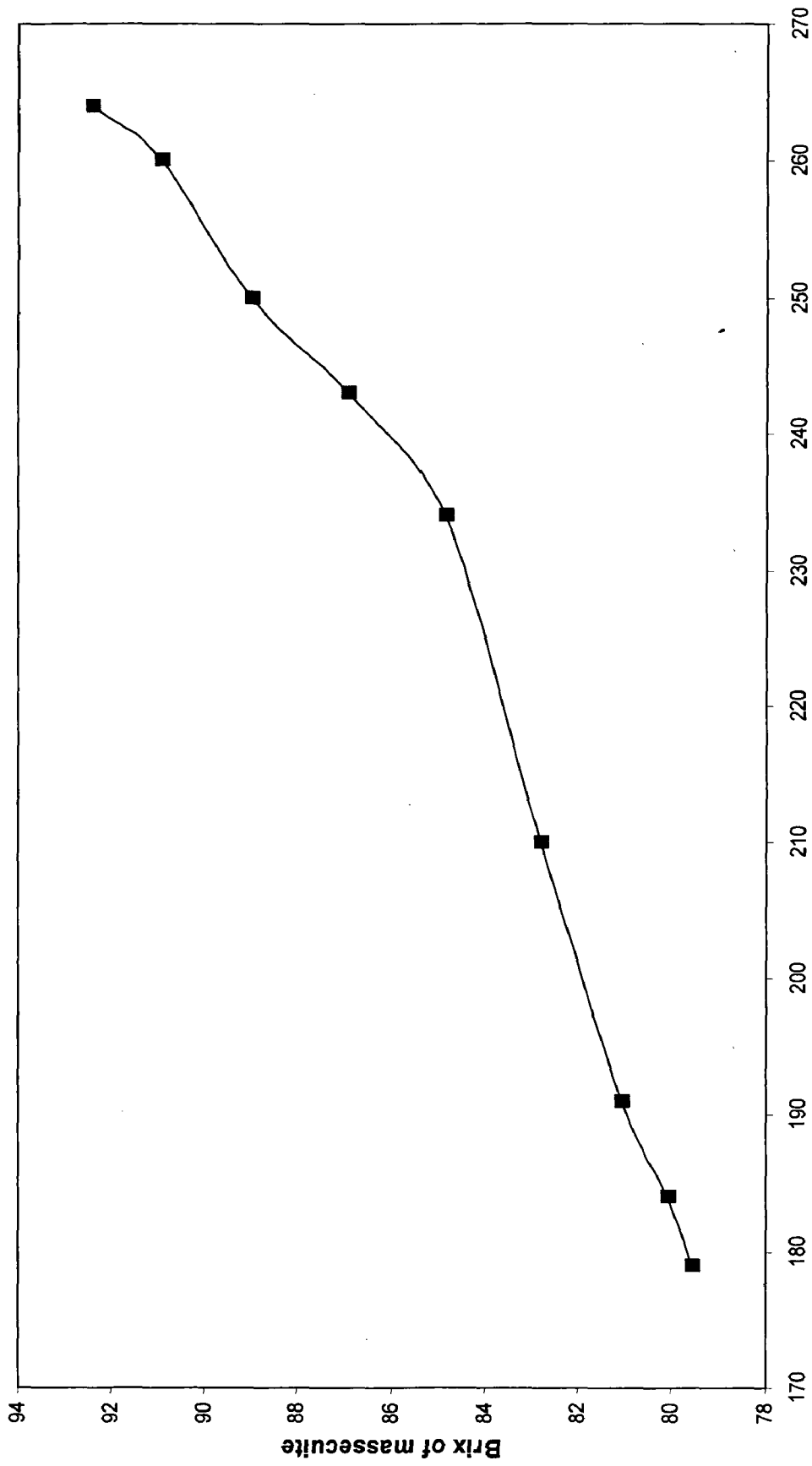


Fig. 5.9 Effect of crystal mean aperture on brix of massecuite

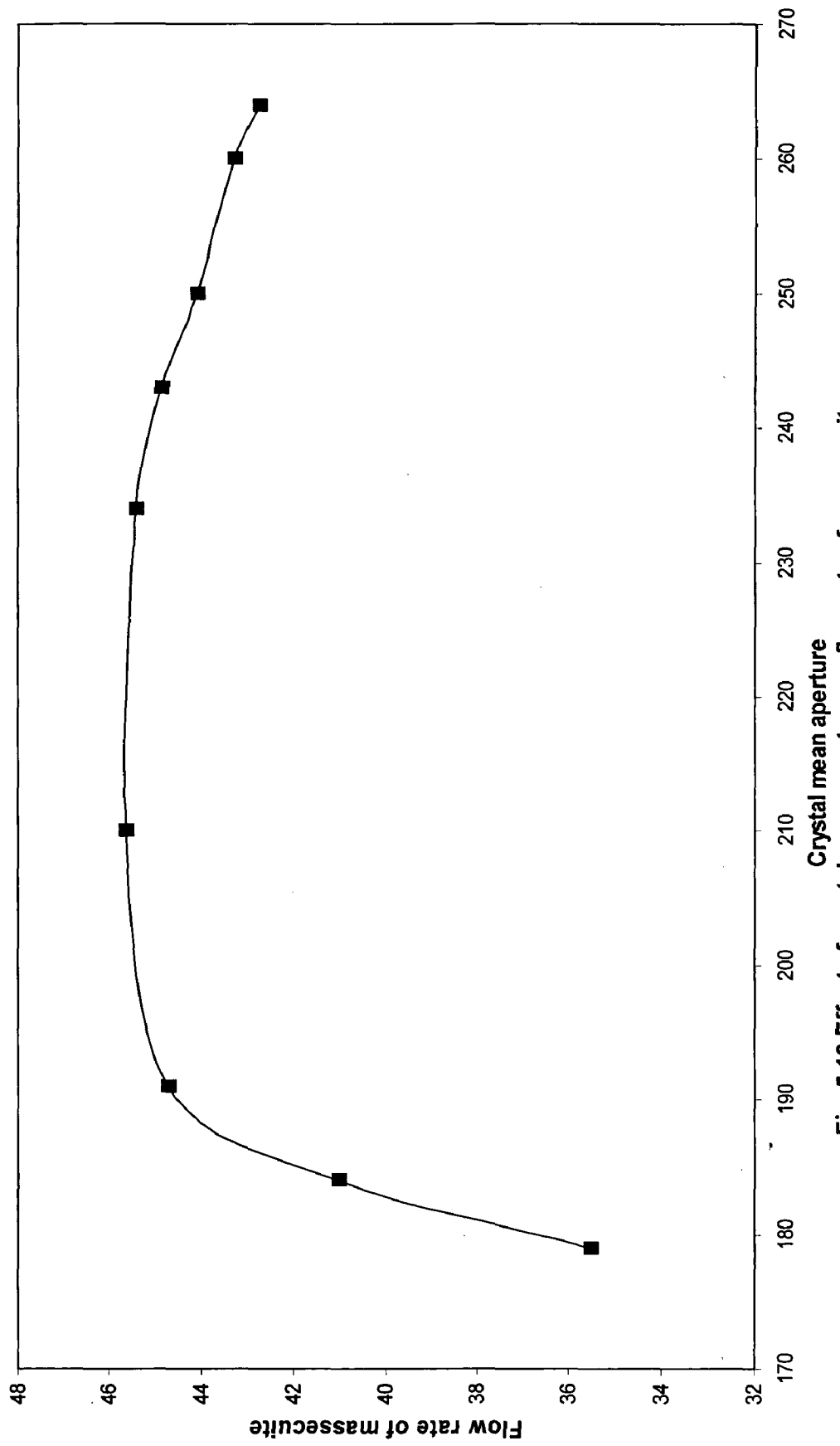


Fig. 5.10 Effect of crystal mean aperture on flow rate of masseccuite

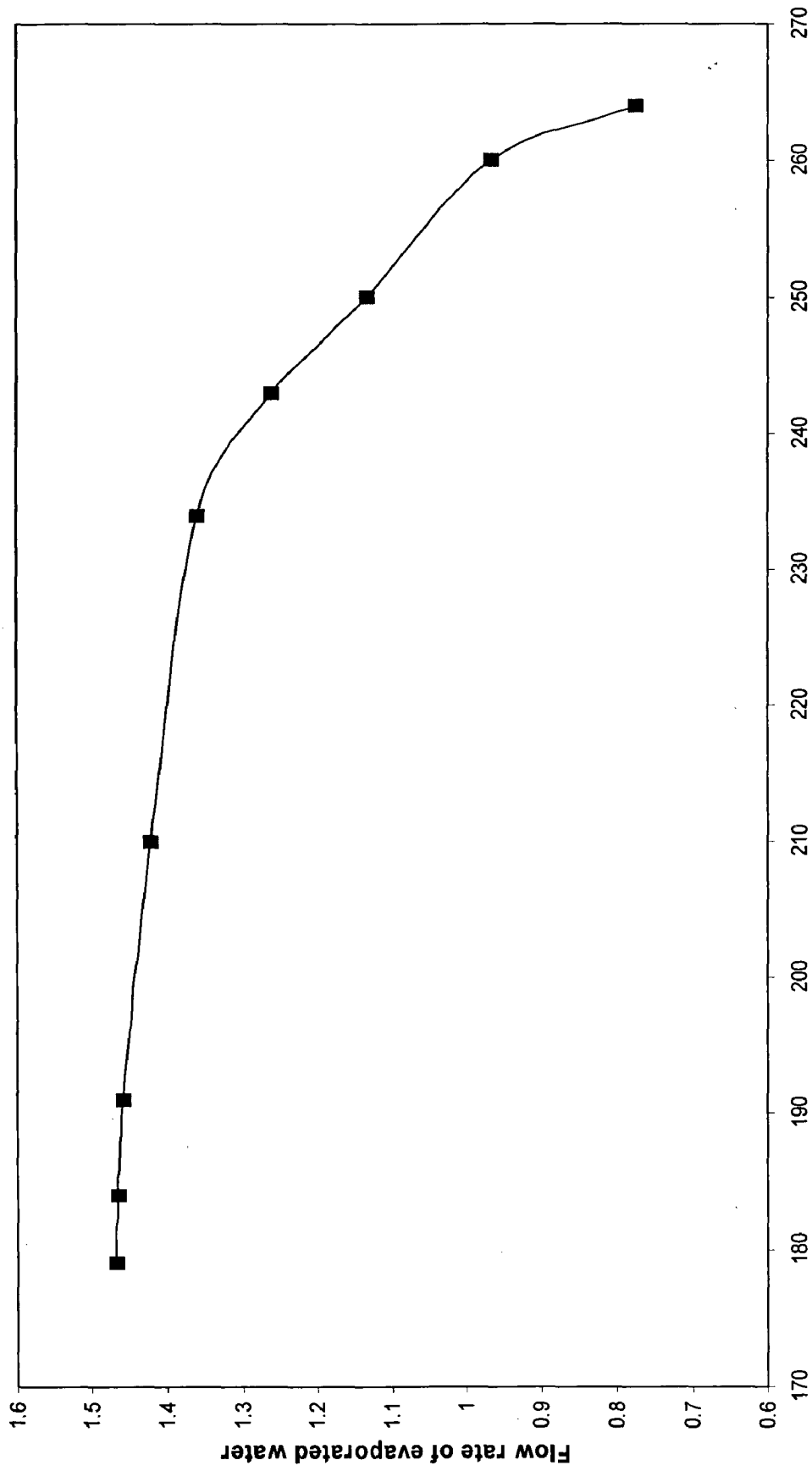


Fig. 5.11 Effect of crystal mean aperture on flow rate of evaporated water

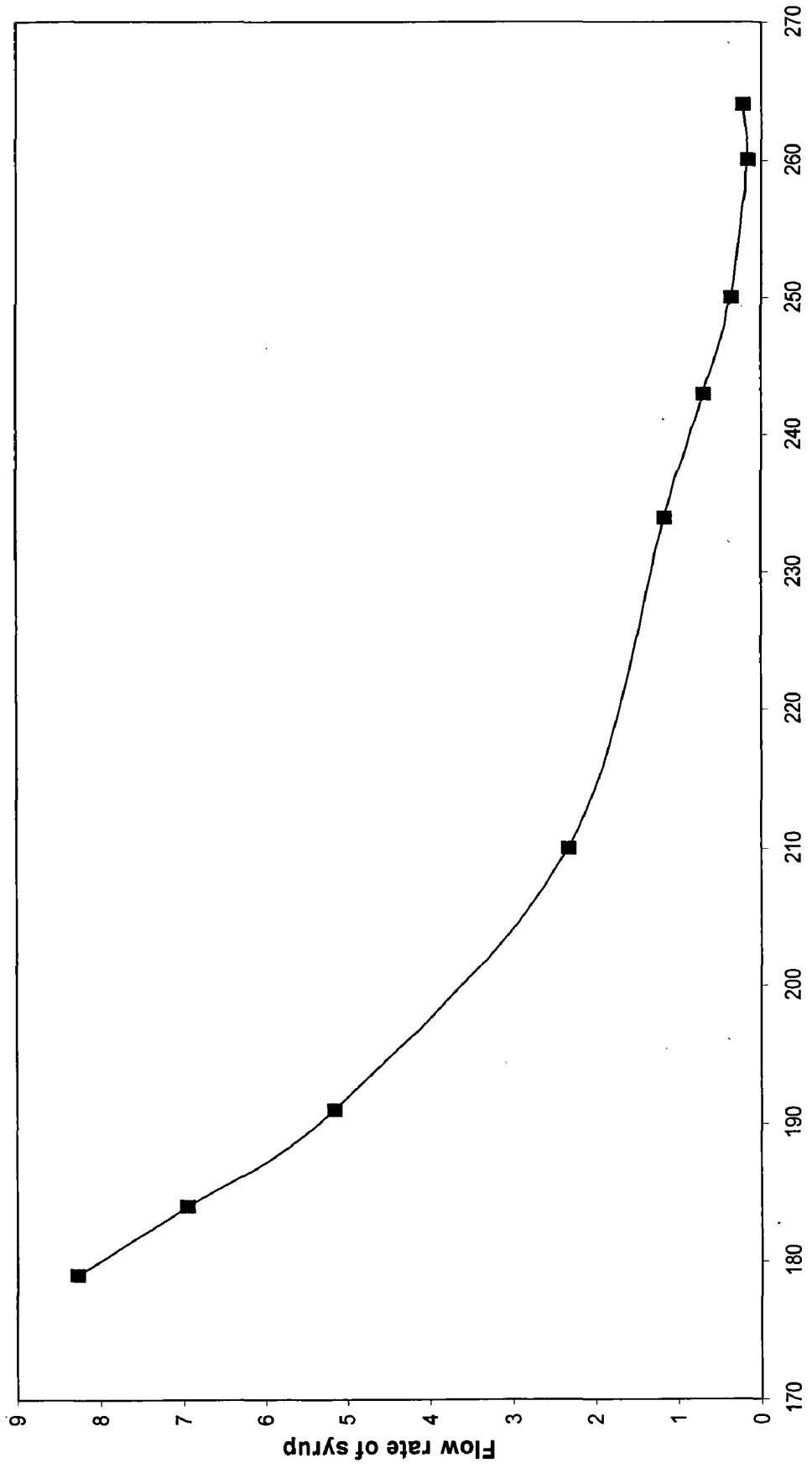


Fig. 5.12 Effect of crystal mean aperture on flow rate of syrup

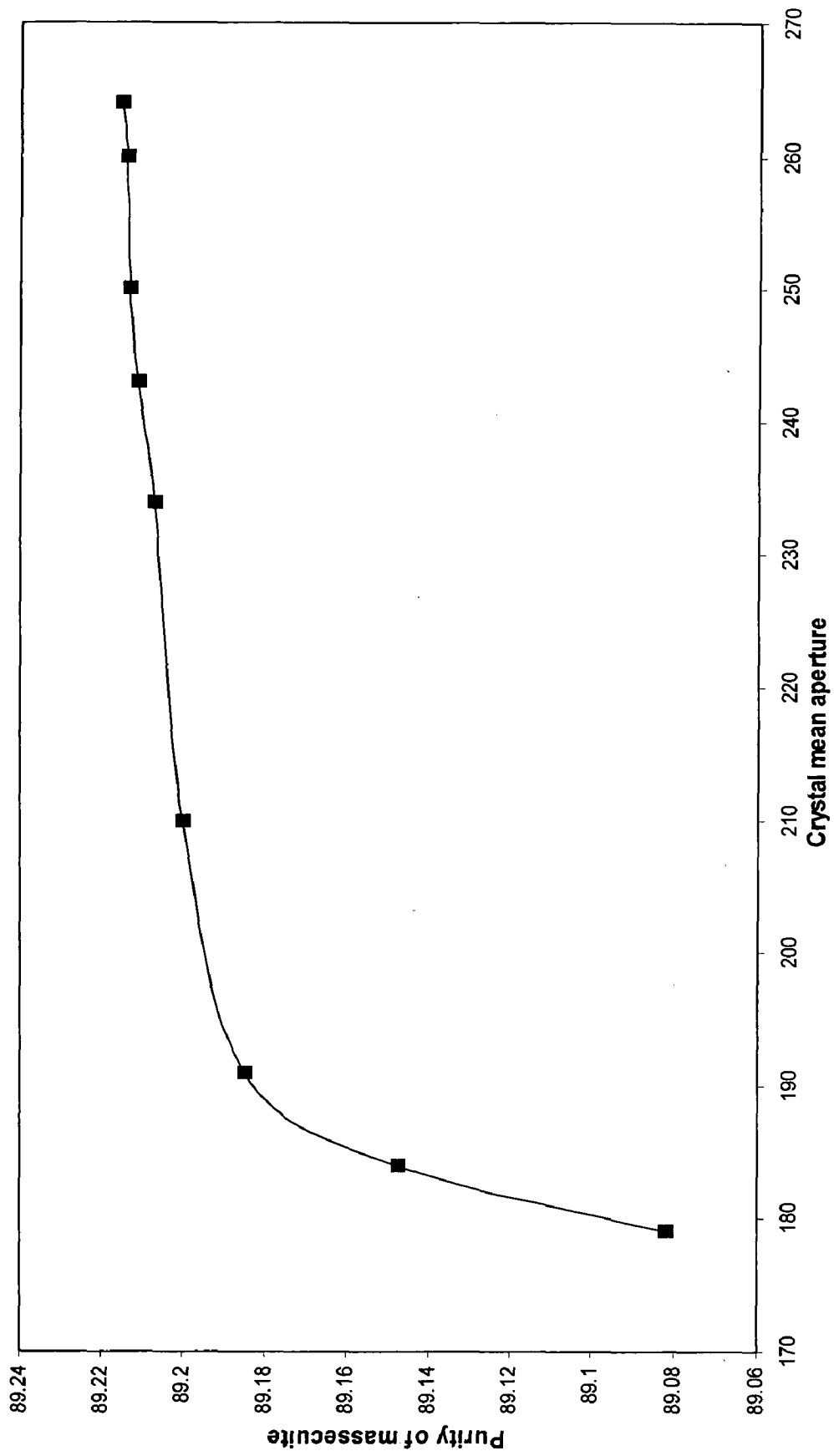


Fig. 5.13 Effect of crystal mean aperture on purity of massecurite

5.2.3 EFFECT OF PURITY OF MASSECUITE

Fig. 5.14 shows the effect of purity of massecuite on the flow rate of massecuite. It shows that in the starting flow rate of massecuite increases with increase in the purity of massecuite, but after that flow rate of massecuite decreases with increase in the purity of massecuite, because as the purity of massecuite increases it means crystal content also increases, due to increases in crystal content flow rate of massecuite decreases.

5.2.4 EFFECT OF BRIX OF MASSECUITE

Fig. 5.15 shows the effect of brix of massecuite on the flow rate of massecuite. It shows that in the starting flow rate of massecuite increases with increase in the brix of massecuite, but after that flow rate of massecuite decreases with increase in the brix of massecuite.

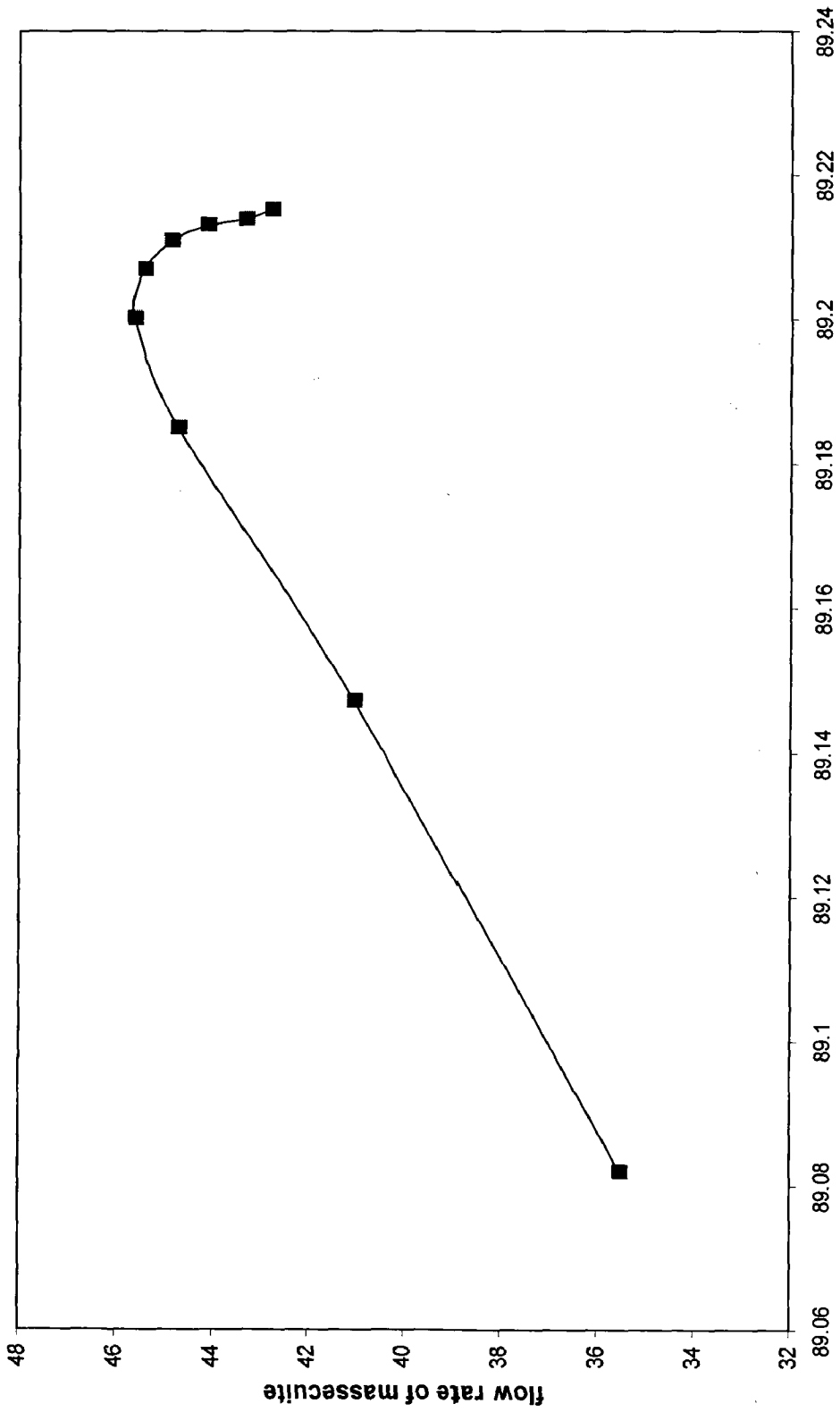


Fig. 5.14 Effect of purity of masseците on flow rate of masseците

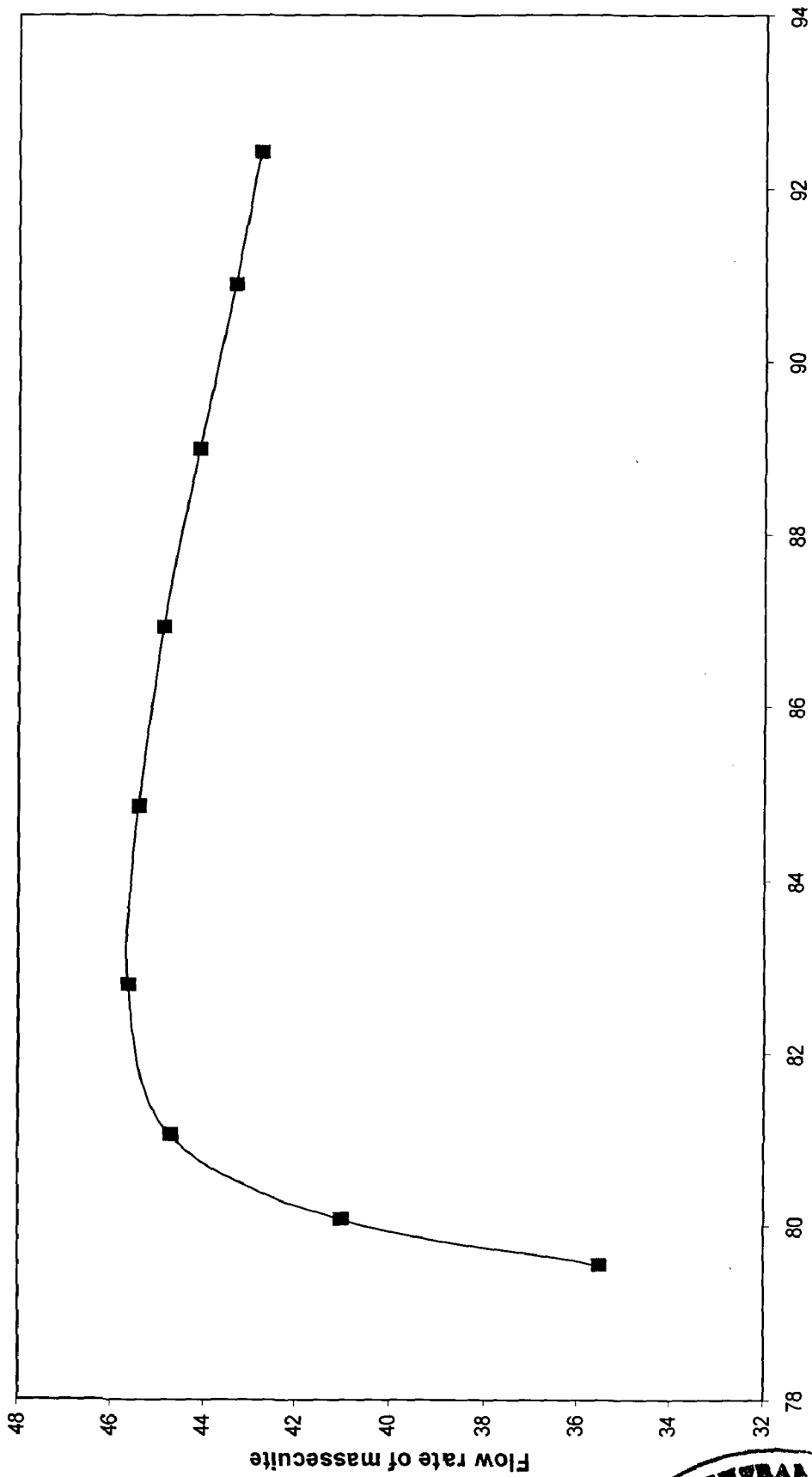
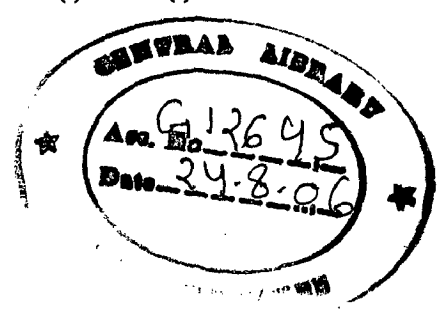


Fig. 5.15 Effect of brix of massecuite on flow rate of massecuite



CONCLUSIONS AND RECOMMENDATIONS

The testing of the model and the computational technique helps us to draw the following conclusions:

1. This model has been satisfactory in simulating a vacuum crystallizer for the system under consideration. The results have been found to agree well with those of Bounahmidi, T. *et al* (2001).
2. The crystal content and crystals mean aperture graphs match excellently, within the error range of 4 percent for each.
3. This model and computational technique have also been successful in determining the effect of the various parameters in vacuum crystallizer, with the results which could be satisfactory.
4. The system has been found most sensitive to crystal content, brix of massecuite and purity of massecuite changes of the feed. The major effect of these being due to crystal content changes.
5. It has been found that the error for each parameter is also less as compared to the error of Bounahmidi, T. *et al* (2001) model.

Although this model and computational technique has been able to simulate the vacuum crystallizer within satisfactory limits, still many of the simplifications which exist now could be rectified and are henceforth being recommended here for further analysis:

1. Here only iterative method has been used for the solution of the model equations. Other techniques could also be used for the solution of the model equations.
2. It is assumed that there is no nucleation; we can solve the model by taking nucleation phenomena in consideration.

Appendix A

Operating Conditions of the Plant

Table A-1 Characteristics of vacuum pan

Number of compartments	9
Working volume	60 m ³
Heat transfer surface area	538 m ²
Vacuum pressure	620 mmHg

Table A-2 Characteristics of materials

	Syrup	Massecuite
Mass flow rate (t/h)	43.10	41.77
Brix (wt %)	65.86	92.42
Purity (wt %)	89.55	89.84
Crystal content (wt %)	-----	49.78
Temperature (°C)	70.0	75.0

Table A-3 Measured data on the industrial plant

Compartment	B _m (wt %)	P _m (wt %)	Cr (wt %)	MA (μm)
1	79.56	88.35	11.12	190
3	81.05	87.95	13.18	200
5	84.85	88.72	27.74	220
7	89.00	89.59	40.77	243
9	92.42	89.84	49.78	271

Appendix B

RESULTS OF THE BOUNAHMIDI'S MODEL

Table B-1 Simulation results

Compartment	P _m (wt %)	F _s (t/h)	F _v (t/h)	Cr (wt %)	MA (μm)
1	89.08	8.28	1.47	11.60	181
2	89.15	6.95	1.47	11.37	183
3	89.18	5.17	1.46	13.93	194
4	89.20	2.34	1.42	19.81	212
5	89.21	1.15	1.36	27.15	228
6	89.21	0.71	1.26	34.51	241
7	89.21	0.37	1.13	41.42	251
8	89.21	0.16	0.97	47.41	258
9	89.21	0.23	0.78	51.87	263

Appendix C

RESULTS OBTAINED FROM MODEL SIMULATION

Table C-1 Simulation results obtained for the industrial crystallizer

Compartment	F_m (t/h)	P_m (wt %)	F_s (t/h)	F_v (t/h)	Cr (wt %)	MA (μm)
1	35.53	89.0819	8.28	1.469	11.88	179
2	41.02	89.1472	6.96	1.466	11.00	174
3	44.73	89.1850	5.17	1.460	12.82	191
4	45.64	89.2000	2.33	1.423	19.22	210
5	45.44	89.2068	1.16	1.363	27.99	234
6	44.88	89.2109	0.70	1.263	35.41	243
7	44.11	89.2129	0.36	1.133	40.34	250
8	43.31	89.2139	0.17	0.968	48.06	260
9	42.76	89.2151	0.23	0.778	50.62	264

Appendix D

MODEL MEAN ABSOLUTE ERROR

Table D-1 Model mean absolute error for the operating variables

Parameters	Mean absolute error (%)
Cr (wt %)	3.19
MA (μm)	1.50
F _v (t/h)	0.19
F _s (t/h)	1.31
P _m (wt %)	0.003

Appendix E

MATLAB PROGRAM FOR SIMULATION

```
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%% THIS PROGRAM WILL SIMULATE THE MODEL FOR THE INDUSTRIAL VACUUM
%% CRYSTALLIZER
%% THE MODEL WILL BE SOLVE USING THE ITERATIVE METHOD
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
CLC
Format long
fm = 28.72;          %% FLOW RATE OF MASSECUITE          (T/H)
pm = 88.97;         %% PURITY OF MASSECUITE          (WT %)
cr = 14.70;         %% CRYSTAL CI=ONTENT          (WT %)
pc = 98;            %% PURITY OF CRYSTALS          (WT %)
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%% ITERATION STARTS FOR EACH COMPARTMENT
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
for i=1:1:9
t = [14.05/60, 12.2/60, 11.25/60, 11.13/60, 11.32/60, 11.60/60,
11.94/60, 12.29/60, 12.56/60];
bxm = [79.44,79.56,80.08,81.05,82.80,84.85,86.94,89.00,90.90,92.42];
          %% BRIX OF MASSECUITE          (WT %)
W = fm*1000*(t (i))*(1-(bxm (i)/100));
          %% WATER IN VACUUM CRYSTALLIZER          (KG)
S = fm*1000*t (i)*(((bxm (i)*pm)/10000)-((cr*pc)/100));
          %% SUCROSE IN VACUUM CRYSTALLIZE          (KG)
I = fm*bxm(i)*t(i)*(100-pm)*1000/10000;
          %% IMPURITIES IN VACUUM CRYSTALLIZER (KG)
p1 = 7.418;         %% [mm/ (h.OS)]
p2 = 0.04;          %% [OS]
p3 = -1.75;         %% UNITLESS
rs = 0.1;
a = 1;
T = 65;            %% TEMPERATURE
(DEGREE C)
p5 = 0.011+ (0.00046*T);
```

```

p6 = 0.67+(0.0021*T)-(0.07*(rs/a));
p7 = 0.54+(0.0049*T);
sc = p5*(I/W) +p6+ (1-p6)*exp (-p7*(I/W));
%% SATURATION COEFFICIENT
B = (0.7533+0.00225*(T-65))/((1-(0.7533+0.00225*(T-65))));
ss = (S/W)/ (sc* B); %% SUPERSATURATION OF SUCROSE
os = ss-1; %% OVERSATURATION
OSb = 0.043;
E = 62.76-.8368*(T-60); %% [KJ/g mol]
trm = inv (T+273.16)-inv (333.16);
kt = p1*exp (p3*I/W)*exp ((-E/8.314)*trm);
%% [mm/(h.OS)]

if (os<0)
g = 5*kt*os;
elseif(os>0)&&(os<(1.5*p2))
g = (kt*os)/3;
else
g = kt*(os-p2);
end
G = g/1000000; %% LINEAR CRYSTAL GROWTH RATE m/h
p = 1580; %% DENSITY OF SUCROSE CRYSTALS KG/M3
ka = 5.02; %% SURFACE SHAPE FACTOR
kv = 0.75; %% VOLUME SHAPE FACTOR
N = 4*(2.71^11); %% NUMBER OF CRYSTALS
rc = 3*p*kv*G/ka; %% CRYSTAL GROWTH RATE KG/ (M2 h)
X = (ka*(N^(1/3)))/((p*kv/1000)^(2/3));
d =
[191.1032,213.9344,254.6667,283.0189,310.0707,314.2241,314.0704,317.331
2,315.2866];
u1 = d(i)*t(i);
u2 = 2*d(i)*u1*t(i);
u3 = 3*d(i)*u2*t(i);
u4 = 4*d (i)*u3*t (i);
MA = u4/u3; %% CRYSTALS MEAN APERTURE M
q= [28.72; 14.7; 18.89; 88.97; 1.41];
x = [1, 1.07, 1.27, 1.53, 1.45, 1.25, 1.12, 1.17, 1.04];
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%% ITERATION STARTS FOR THE CALCULATION OF FLOW RATE OF MASSECUITE

```

%%

for Fm=1:0.01:50

s=t (i);

k=cry(X, rc, pc, fm, cr, s, Fm);

k1 = k*x (i);

bx_s = 65.86; %% BRIX OF SYRUP (WT %)

ps = 89.55; %% PURITY OF SYRUP (WT %)

a (1) = bx_s*ps;

b (1) = -Fm*bx_m (i+1);

c (1) = -fm*bx_m (i)*pm;

a (2) = (bx_s*(100-ps)/10000);

b (2) = Fm*bx_m (i+1)/10000;

c (2) = -((fm*bx_m(i)*(100-pm)/10000)-(Fm*bx_m(i+1)/100));

Z = [a (1), b (1); a (2), b (2)];

C = [c (1); c (2)];

Sol=Z\C;

fs₁ = sol (1); %% FLOW RATE OF SYRUP (T/H)

pm₁ = sol (2); %% PURITY OF MASSECUITE (WT %)

T_m = 75; %% TEMPERATURE OF MASSECUITE (DEGREE C)

T_s = 70; %% TEMPERATURE OF SYRUP (DEGREE C)

Fv₁ = fm*(1-(bx_m(i)/100))+fs₁*(1-(bx_s/100))-Fm*(1-(bx_m(i+1)/100));

 %% FLOW RATE OF EVAPORATED WATER (T/H)

hm = ((-0.0324*((bx_m(i)*pm/100)^2)+4.6027*(bx_m(i)*pm/100)-167.44)+(-0.022*(bx_m(i)*pm/100)+4.1084)*T_m)*1000 ;

 %% ENTHALPY OF MASSECUITE KJ/ton

hm₁ = ((-0.0324*((bx_m (i+1)*pm₁/100) ^2) +4.6027*(bx_m (i+1)*pm₁/100)-167.44) + (-0.022*(bx_m (i+1)*pm₁/100) +4.1084)*T_m)*1000;

 %% ENTHALPY OF MASSECUITE KJ/ton

hs = ((-0.001*((bx_s*ps/100) ^3) + 0.1744*((bx_s*ps/100) ^2)-9.9213*(bx_s*ps/100) +177.11) + (-0.0241*(bx_s*ps/100) +4.2606)*T_s)*1000;

 %% ENTHALPY OS SYRUP KJ/ton

U = (2136.4*T_m)/bx_m(i+1);

 %% HEAT TRANSFER COEFFICIENT KJ/ (h m² OC)

A_h = 538; %% HEAT TRANSFER SURFACE AREA m²

T_{vc} = 100.21; %% TEMPERATURE OF HEATING STEAM (DEGREE C)

h_v = 2259.29; %% HEAT OF VAPORIZATION KJ/Ton

```

hc=[-3.407249474862218e+005,-4.378113336444934e+005,-
4.189113439958620e+005,-2.663244477384184e+005,-
1.336697169716074e+005,-7.704536024641332e+004,-
5.648537577884220e+004,-4.694842624669449e+004,-
4.360877144195131e+004];
fx= (fm*hm+fsl*hs-Fm*hm1-Fv1*hv+U*Ah*(Tvc-Tm))-(X*((k1*s*Fm/100) ^
(2/3))*rc)*hc (i);
if (abs (fx) <1)
    y = [Fm; k1; fsl; pm1; Fv1; MA]
        break
else
    continue
end
end
fm = Fm;
cr = k1;
pm = pm1;
end
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%% THIS IS A FUNCTION FOR THE CALCULATION OF CRYSTAL CONTENTS IN EACH
%% COMPARTMENT
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
Function [c] = cry(X, rc, pc, fm, cr, s, Fm)
cri = 1;
for z = 1:1:100
fcri = X*((cri*Fm*s/100) ^ (2/3))*rc/1000-(cri*Fm*pc)/ (100) +
((fm*cr*pc)/100);
gcricri = (2/3)*X*rc*((cri*Fm*s/100) ^ (-1/3))*Fm*s/100000-(pc*Fm)/ (100);
cri = cri-(fcricri/gcri);
end;
c = cri;

```

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