Improvement of Safety Characteristics of Stirred Reactors (*SR*)

VisiMix® Approach to Inherently Safer Design of SR

Y. Nekhamkin, L. Braginsky, Y. Kokotov

VisiMix Ltd. 8 Hamarpe Street 7th Floor, Bynet Bldg.,

Jerusalem 97774 Israel

email: info@visimix.com

Content

- Introduction
- Motivation
- Background – VisiMix® Main Features
- VisiMix® Applications for Inherent Safer Design and/or Inherent Safer Technology (***ISD***/***IST***) of Stirred Reactors (***SR***)
  - ✓ Tackling SR Safety Problems at the Design Stage
  - ✓ Tackling SR Safety Problems during Operations
  - ✓ Tackling SR Safety Problems for Accident/Incident Investigation
- Conclusions

Introduction:

1. A set of the VisiMix® software codes was developed for *process engineers* as a *tool for solving a wide range of technological problems in stirred reactors.*
2. VisiMix® gained recognition because *it provides an adequate and a complete description of processes in stirred reactors accounting corresponding equipment configuration based on reliable models verified in practice* [*www.visimix.com* and *CIO Review, page 50: VisiMix One of 2014’s 20 Most Promising Chemical Tech Solution Providers* in *http://magazine.cioreview.com/Jan-2015/Chemical/*].

Introduction (cont'd):

Customers and Markets

Total > 200 customers

Introduction (cont’d):

3. VisiMix® **potential** enables to enhance its field of application and use it to improve safety characteristics of the considered stirred reactor that renders the VisiMix® an efficient tool for safety experts too in the following instances:

(i) at reactor and/or process design stage;

(ii) during operation stage;

(iii) for accident/incident investigation.

<u>Motivation</u>

1. **The stirred reactors operations are followed by numerous accidents.**

   Stirred reactors (***SR***) belong to the key type of equipment used in all branches of the chemical process industry (***CPI***). The SR operations are followed by ~14% of all accidents in Chemical Process Industry with 71% of them related to batch/semi-batch reactors operations. These accidents are caused by different reasons, namely:

   * inadequate process analysis on heat transfer (~23%),
   * reaction problems (~23%),
   * process contamination (16%), etc.

Kamarizan Kidam and Markku Hurme, Analysis of equipment failures as contributors to chemical process accidents. Process Safety and Environmental Protection, 91 (2013), pp. 61-78].

Motivation:

2. The Inherently Safer Design/Technology (*ISD / IST*) concept

[*T.A. Kletz and P. Amyotte, Process plants. A handbook for inherently safer design. 2nd Ed., Taylor CRC Press, Boca Raton. Fl.(2010)*]

This concept requires that the process engineer challenge is

(i) "to identify ways to eliminate the hazards associated with the process, rather than to develop add-on barriers to protect people from these hazards";

(ii) to use "appropriate analytical and decision making tools to select him the best overall process alternative, considering all of the hazards".

[*D.C. Hendershot, Inherently Safer chemical process design. Journal of Loss Prevention in the Process Industries, Vol.10, (1997), pp. 151…157*].

Background – VisiMix® Main features

1. **The VisiMix® is a set of software tools for simulation and technical calculation of mixing related processes developed for *process engineers* as a universal tool for solving a wide range of technological problems.**

VisiMix® includes the following software tools [www.visimix.com] :

* VisiMix Turbulent® for turbulent flow regime (low viscosity flow),
* VisiMix Laminar® for laminar flow regime (high viscosity flow and flow of non-Newtonian liquids),
* VisiMix Different Impellers® for simulating mixing devices with different impellers (to be used with VisiMix Turbulent®),

- VisiMix RSDE® for simulation of rotor stator dispersers,
- VisiMix Pipe-Line® for hydraulic calculations for low and high viscous and non-Newtonian liquids in plant pipe lines,
- VisiMix Excel® that integrates VisiMix reports in a standard Excel worksheet.

Background (cont'd)

2. The VisiMix® includes the following Main Unit Operation Topics

(List conforms to *the VisiMix Turbulent*® and *VisiMix Laminar*® software tools)

Process / Unit Operation	Problem and Key Mixing Parameters
1. Basic mixing information	Main mixing characteristics
	Flow dynamics
	Vortex formation
	Turbulence, shear rates and stresses
2. Blending (distribution of a solute)	Mixing time
	Simulation of blending
	Micromixing
3. Liquid-solid mixing:	
3.1. Suspension	Checking "non-settling" conditions
	Distribution of solid phase
3.2. Dissolution of solid	Completeness of dissolution
	Simulation of a dissolution process
	Mass transfer

Background (cont'd)

2. The VisiMix® Main Unit Operation Topics (cont'd)

Process / Unit operation	Problem and Key Mixing Parameters
3.3. Leaching (liquid-solid extraction)	Collisions of particles
	Mass transfer
	Distribution of solid phase
3.4. Crystallization	Uniformity of mother solution
	Mixing parameters affecting nucleation and growth of crystals
3.5. Heterogeneous liquid-solid reaction	Mass transfer characteristics
4. Liquid-liquid mixing:	
4.1. Emulsification	Completeness of emulsification Drop size distribution
	Mixing parameters affecting emulsification
4.2. Liquid extraction and heterogeneous liquid-liquid reaction	Mass transfer characteristics
	Mixing parameters affecting liquid extraction and reaction

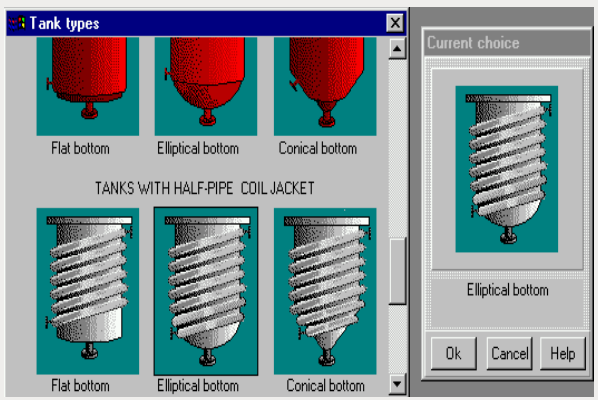
Background (cont'd)

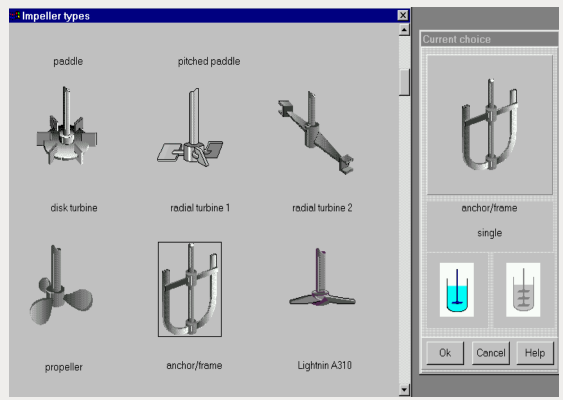
2. The VisiMix® Main Unit Operation Topics (cont'd)

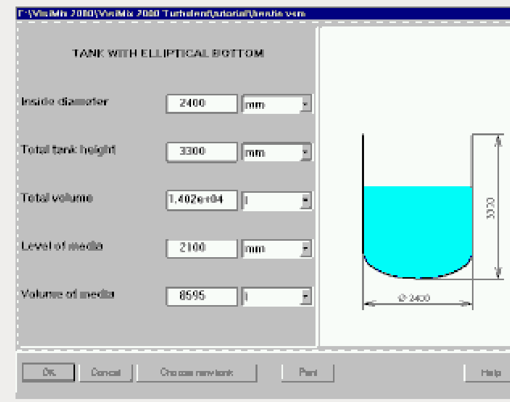
Process / Unit Operation	Problem and Key Mixing Parameters
5. Single phase chemical reaction . Batch, Semi-batch and Continuous flow reactors	Local concentration of reactants
	Non-uniformity of mixing in reactor
	Selectivity of reaction
	Dynamic characteristics
	Approach to "perfect mixing"
6. Mechanical reliability	Stresses in dangerous cross-section
	Shaft vibration
7. Temperature-dependent reaction. Batch, Semi-batch and Continuous flow reactors	A comprehensive set of heat transfer characteristics Simulation of thermal regimes
8. Thermal safety	Analysis of runaway process
	Prediction of media overheating/overcooling

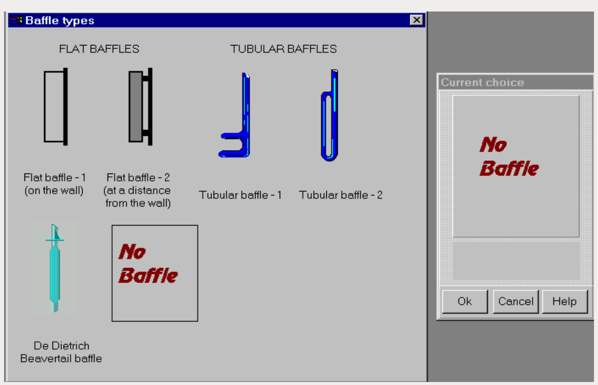
Background (cont'd)

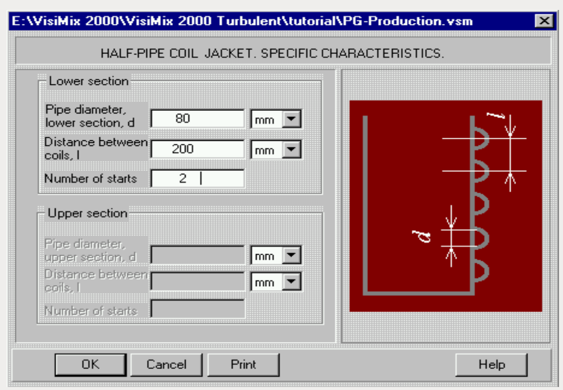
3. The Visimix® codes perform simulation of different processes with ***respect to real equipment design*** and ***process regime parameters***. Selection of equipment types and entering their and process parameters is effected by means of the ***simple user-friendly graphic user interface*** .

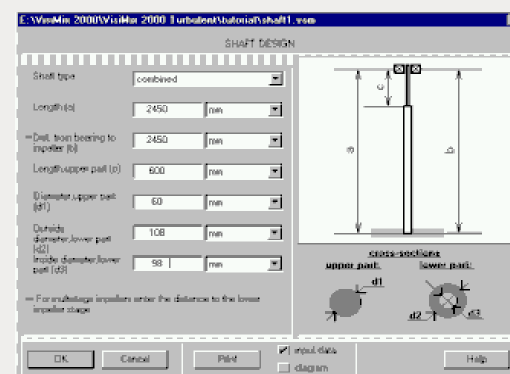












Background (cont'd)

4. The Visimix® simulation capabilities are sustained by means of

- **build-in *databases with properties of applied materials* and**
- ***HELP system with enhanced technical information* that endows it with properties of the reference source.**

Background (cont'd)

5. The Visimix® has a property of an ***expert system* that analyses initial data and calculation results** and **issues *warning messages* whenever the input results in unacceptable process course.**

Hence, messages system is a mean to define safety range of the basic process parameters. Typical *VisiMix* ® messages are depicted below.

Message	Possible reason	Hazard potential
Mixing power is too high for your drive	High viscosity or density of media. Incorrect drive selection	Possible unexpected stop of mixing. Shaft breaking (check Shaft design with Visimix).
Complete suspension is questionable	Big particle size for given mixing system. Too high concentration of solid phase.	Increase of bending moment and shaft/sealing breaking. Plugging of outlet from reactor. Hot spot formation.
Centrifugal separation of emulsion is expected. Addition baffles is advisable	Incorrect design for liquid-liquid mixing reactor	Decrease in dispergation ability of impeller and interface area Unpredicted reaction and mass transfer rate

Background (cont'd)

5. The Visimix® has a property of an *expert system*…

Message	Possible reason	Hazard potential
Vortex reaches impeller! Gas insertion from surface and shaft vibration are possible	Too intensive mixing. Not enough baffles resistance. Impeller too close to surface.	Mechanical breaking. Unpredicted way of reaction. Foam formation. Unwanted oxidation.
After *XXX* sec have elapsed, the temperature falls outside the indicated range of process temperature	Weak heat transfer system. Too low mixing. Not proper design.	Unexpected way of the process. Agglomeration of solid particles, fouling of wall and damage to the heat transfer. Increase of pressure. Explosion.
This heat transfer agent doesn't correspond to process temperature range	Not proper heat transfer agent selection	Fail of heat transfer

Background (cont'd)

5. The Visimix® has a property of an *expert system*…

Message	Possible reason	Hazard potential
Rotational frequency of the shaft is too close to critical frequency. Vibrations are possible, see SHAFT VIBRATION CHARACTERISTICS	Small shaft diameter Unexpectedly high power	Mechanical breaking
Fluid velocity is too low for efficient mixing	Not proper design	Unpredicted way of the process Damage to the product Plugging of the reactor
Inefficient mixing because of short-circuiting of flow in impeller area. See output parameters "Scheme of main circulation cycles" and "Circulation flow rate", and HELP		
Formation of stagnant zones is expected. For recommendations see HELP, Formation of stagnant zones		

Background (cont'd)

Thus if we recall that *any accident/incident stars from small deviation from the normal process course*, the **VisiMix® message system enables to define ISD/IST range of stirred reactors.**

VisiMix® application for ISD/IST of Stirred Reactors

Example 1. Tackling safety problems at the ***design stage***.

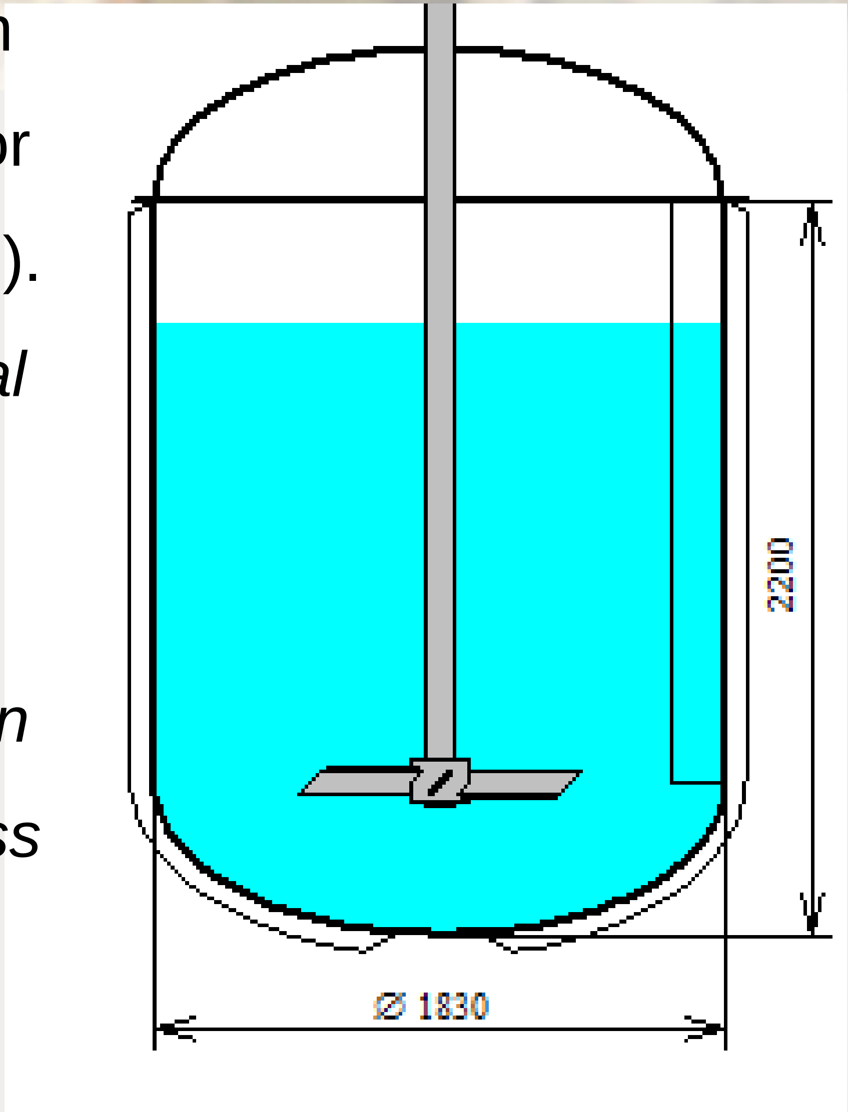
Problem Description. The actual process is performed in two stages:

- **Initial heating** which starts the reaction and. The heating is achieved by *steam at atmospheric pressure* supplied into the jacket,
- **Subsequent cooling** required for removing excessive heat. The cooling is realized with *ordinary water at 20*℃ circulated through the jacket.

VisiMix® application for ISD/IST of SR

Example 1. Tackling safety problems at the ***design stage*** (cont'd).

1. The considered process occurres in a *cylindrical fully baffled* stirred reactor with *pitched paddle impeller* (85 RPM).
2. The process is based on *exothermal catalytic reaction involving solid catalyst.*
3. Aim of the analysis is *to define main process parameters and the process duration.*



<u>VisiMix® application for ISD/IST for SR.</u>

<u>***Example 1***</u> **(Cont'd)**

The considered problem from the safety standpoint raises ***two tasks*** to be solved.

<u>**The *first task***</u> is to define *Just Suspension Speed* (***JSS***) – the minimum rotational velocity of the impeller at which there are no stagnant zones at the bottom.

<u>**The *second task***</u> is to simulate a *second order exothermal reaction* in a stirred batch reactor aiming to avoid *runaway reaction* that takes place when the energy generated by the reaction is greater than the energy removed from the reactor.

Kinetic reaction parameters were partially borrowed from the book *Safety, Health, and Loss Prevention in Chemical Processes. Problems for Undergraduate Engineering Curricula,* (The Center for Chemical Process Safety of the American Institute of Chemical Engineers) pp. 162 - 164, *Problem No. 81.*

VisiMix® application for ISD/IST of SR

Example 1 (Cont'd)

Problem Solution.

Task1. Choosing parameter **Complete/incomplete suspension** in the **Liquid-Solid Mixing** submenu results in warning messages informing the user of possible settling of solid particles every time when settling occurs.

Complete suspension is questionable

or

Partial settling of solid phase may occur

Thus the JSS is the minimum value of rotational velocity which does not result in the warning message. In our case JSS is equal to 72 RPM (less than 85 RPM) with a corresponding message.

Complete suspension is expected

VisiMix® application for ISD/IST …. *Example 1* (Cont'd)

Problem Solution (cont'd).

Task 2. Heat transfer calculations performed for the *first* process stage (initial heating at first cause appearance of a warning message informing that at 443 sec (~7.3 minutes) a runaway reaction had started

After 443 sec have elapsed, the temperature falls outside the indicated range of process temperatures

Note that if instead of steam another heating agent Dowtherm SR-1 is selected the program will send a warning message .

This heat transfer agent doesn't correspond to process temperature range.

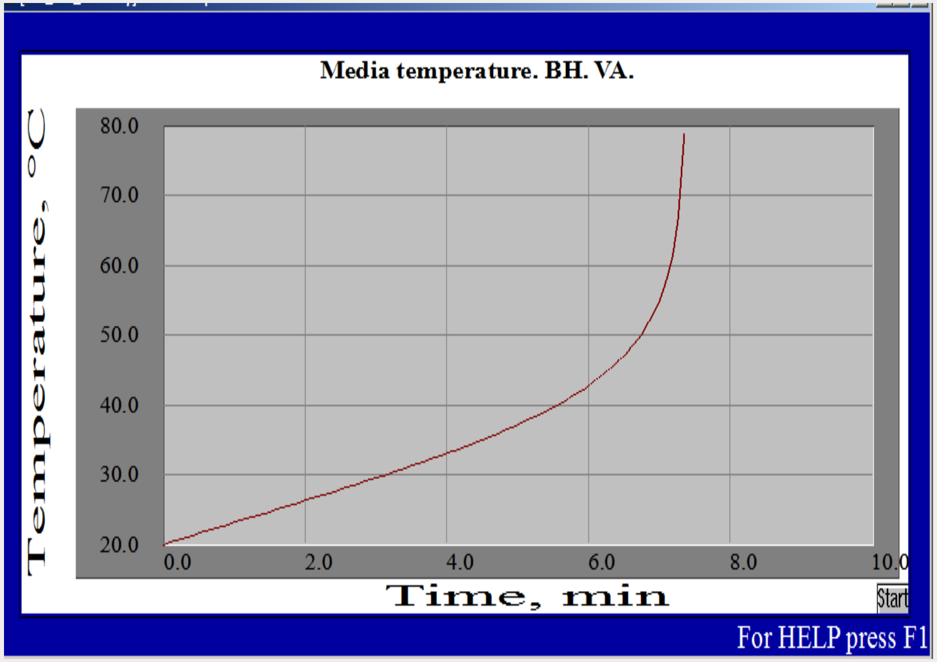
The reason is that an operating temperature of Dowtherm SR-1 is 121°C that is less than the process upper temperature limit (143°C).

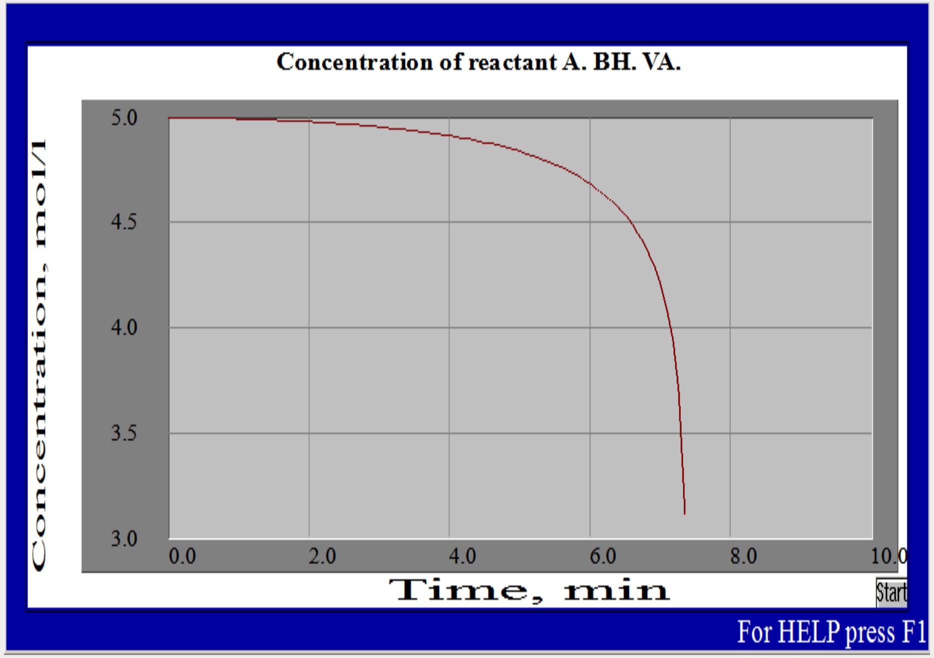
By this means that VisiMix® enables to avoid mistakes caused by a wrong selection of the cooling/heating agent.

VisiMix® application for ISD/IST …. *Example 1* (Cont'd)

Problem Solution (cont'd).

Media temperature and concentration history for the *first* process stage are presented below.



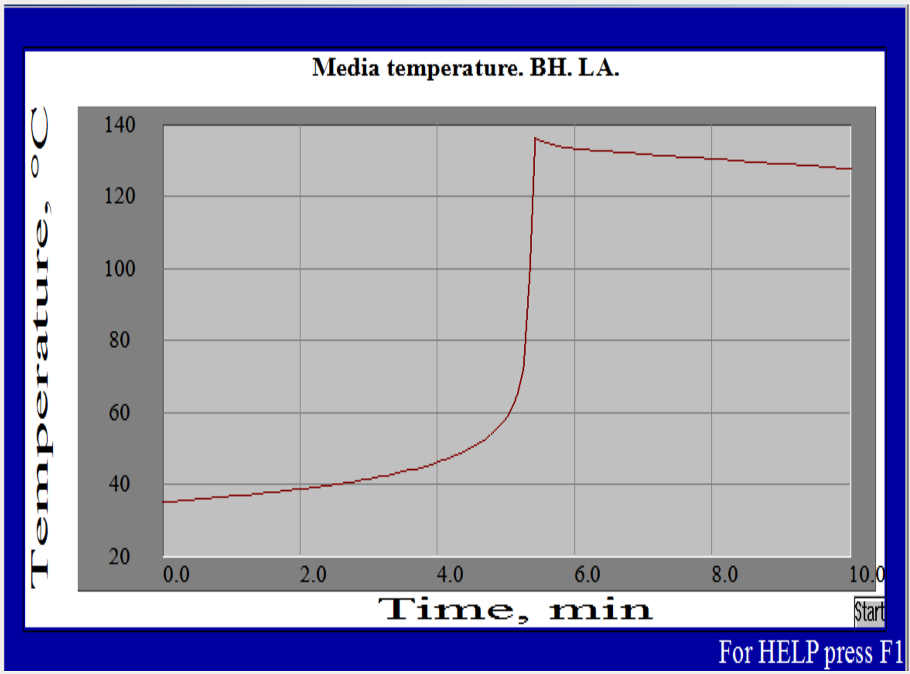


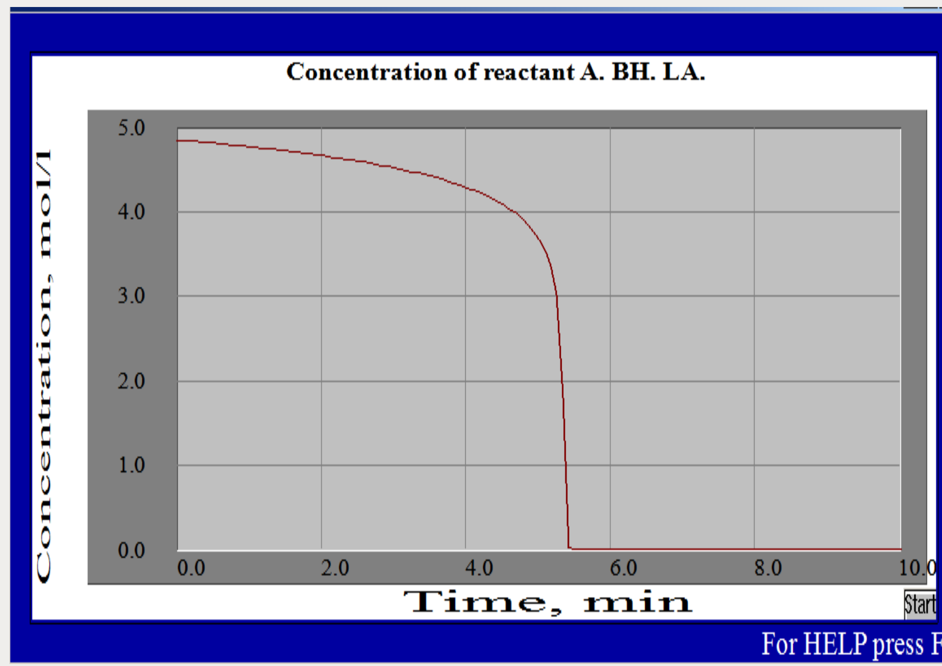
Based on these graphs, the ***first stage duration*** is taken to be limited by ***4.5 minutes*** from the start of the process and initial parameters of the second stage are ***35℃*** and ***4.85 mole/liter***, respectively.

VisiMix® application for ISD/IST …. *Example 1* (Cont'd)

Problem Solution (cont'd).

Results of the heat transfer calculations for the ***second*** stage with initial data defined in the previous slide are presented below





The duration of the cooling stage is about ***5.3 minutes***, and the total reaction time is about ***10 minutes***.

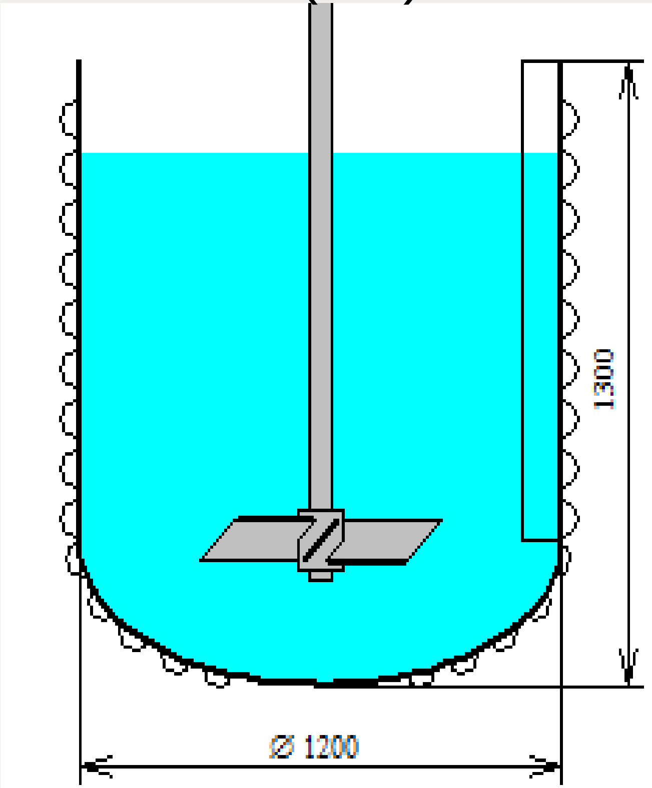
VisiMix® application of ISD/IST …. *Example 2*

Example 2. Tackling safety problems during ***operations***.

Process Description. This example taken from [H. Scott Fogler, *Elements of Chemical Reaction Engineering,* 2nd ed. (Prentice-Hall, Inc.(1992), pp.400-405, (*Examples 8-4 and 8-5*).] considers production propylene glycol (**PG**) by the hydrolysis of propylene oxide (**PO**):

$$PO + H_2O \xrightarrow{H_2SO_4} PG$$

This exothermal reaction takes place at room temperature when catalyzed by sulfuric acid in a 300-gal reactor. The process has an ***operating constraint***: the temperature of the mixture inside the reactor must not exceed 130°F because of the low boiling point of **PO** .



<u>VisiMix® application of ISD/IST …. *Example 2*</u> (Cont'd)

Statement of the Problem.

1. Operation of any reactor over prolonged period causes a ***fouling layer*** in a tank jacket to grow that in turn will lead to increase of *additional thermal resistance* between in-tank media and in-jacket coolant. The last-mentioned affects the temperature regime inside the tank and may cause runaway reaction.

2. The ***fouling growth*** results in a ***rise of the media temperature*** which is usually compensated by increasing, accordingly, the *supply of coolant* into the jacket (the coolant flow rate and the corresponding *pressure head* on the jacket). The latter is usually used in control systems and corresponds to the VisiMix® parameter **Pressure head on the jacket**.

<u>VisiMix® application of ISD/IST …. *Example 2* </u>(Cont'd)

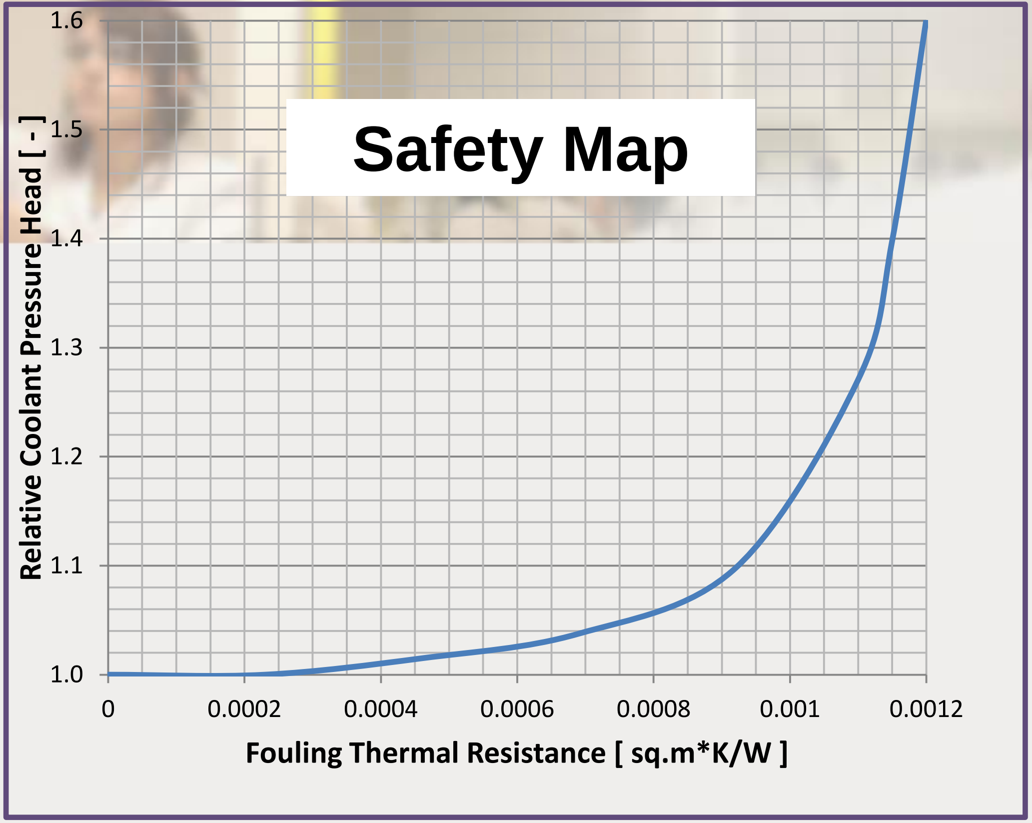
Statement of the Problem (cont'd).

3. Since the ***capabilities of control systems are limited***, it is necessary to *ensure the system to maintain the required pressure head* in the considered case. Based on calculation results, the dependence of the **Relative Coolant Pressure Head** (on the jacket) on the **Fouling Thermal Resistance** ("***Safety Map***" for this process) was displayed graphically. The pressure head values used for this graph correspond to the minimum flow rate of the coolant at which the media temperature does not exceed the allowable limits.

This **Safety Map** enables to check the ability of the control system to provide the required pressure head for compensating temperature rise caused by fouling.

VisiMix® application of ISD/IST … *Example 2* (Cont'd)

The Safety Map shows how the system is approaching the dangerous state when the control system is no longer capable of providing the required cooling and hence is setting the condition for the beginning of a runaway regime.

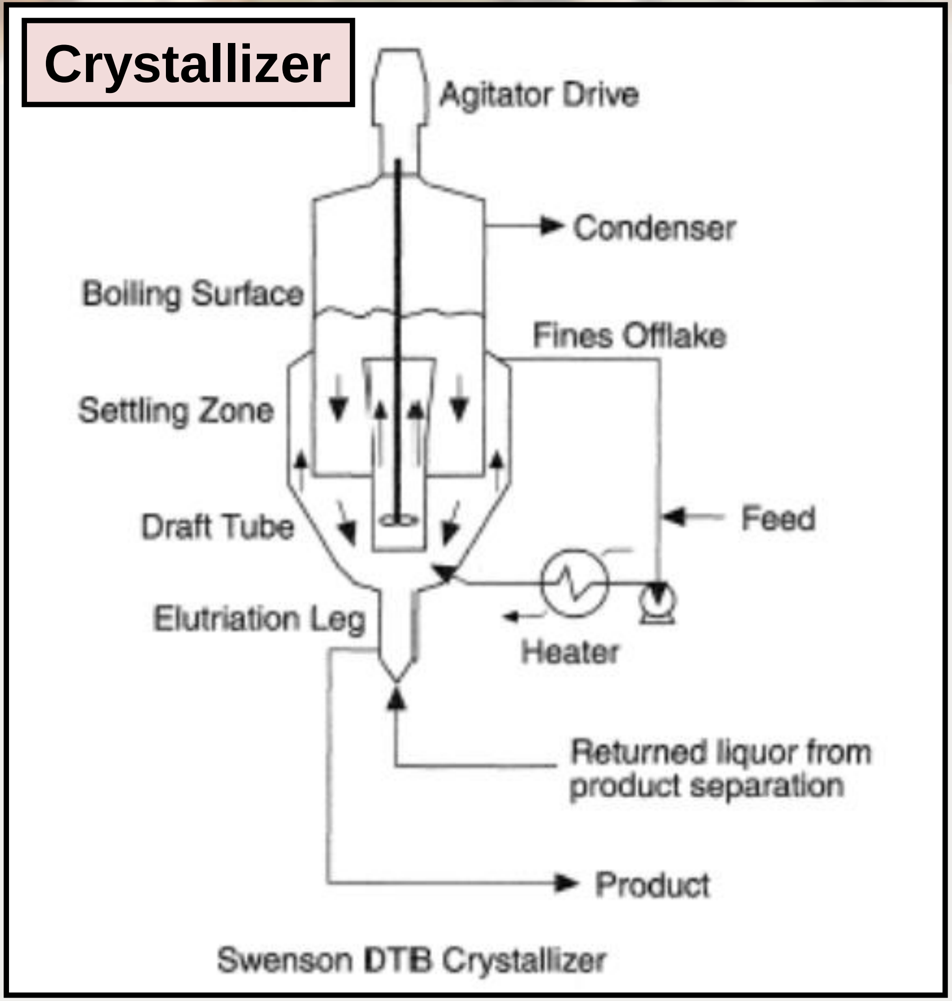


The initial fouling thermal resistance ~0.001 m^2*K/W (for pure service water used as a coolant it corresponds to deposit layers with ~2mm thickness). Thus the VisiMix® enables to define a ***criterion setting an upper limit for the process stoppage.***

VisiMix® application of ISD/IST …. *Example 3*

Example 3. Tackling safety problems for *incident/accident investigation.*

Incident Description. An incident took place in the crystallizer for the phosphoric acid production (its volume > 2000 m^3) equipped with a massive cast impeller with a tip diameter 5.33 m, and mass about 2000kg) and a *draft tube*. The crystallizer design is similar to the one depicted at the left hand side.

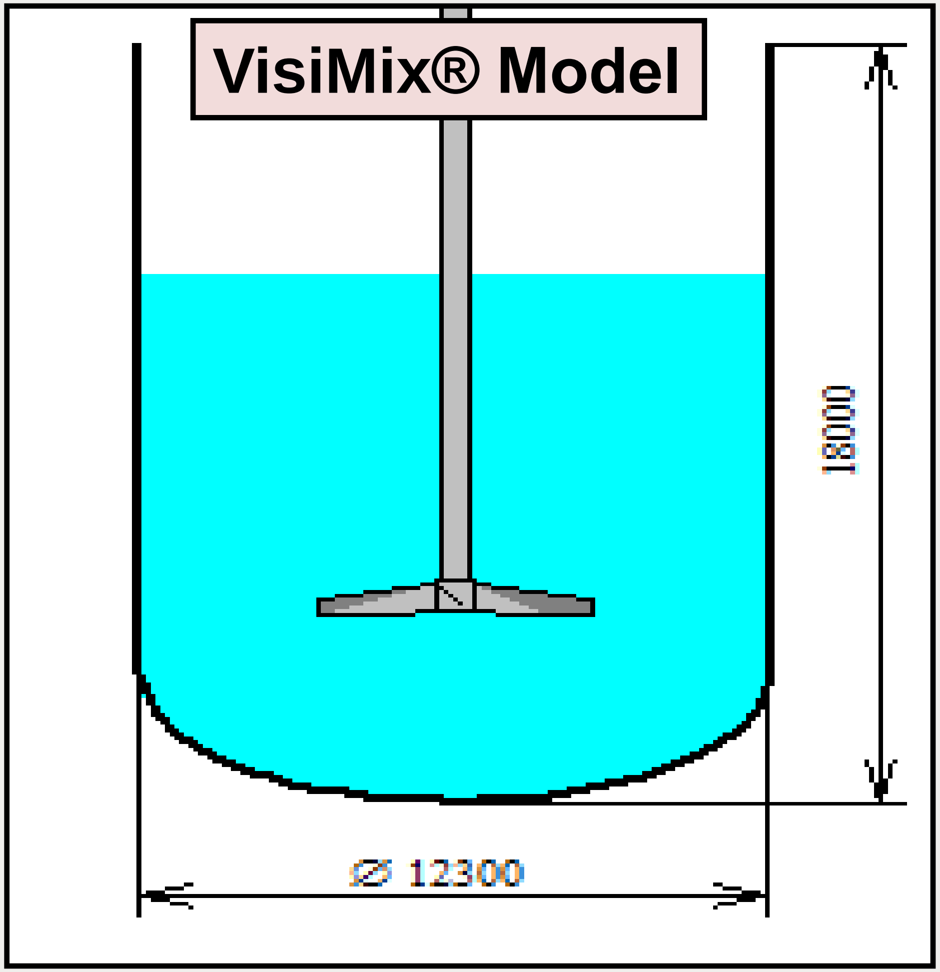


The incident starts shortly after the drive motor startup.

<u>VisiMix® application of ISD/IST …. *Example 3*</u>

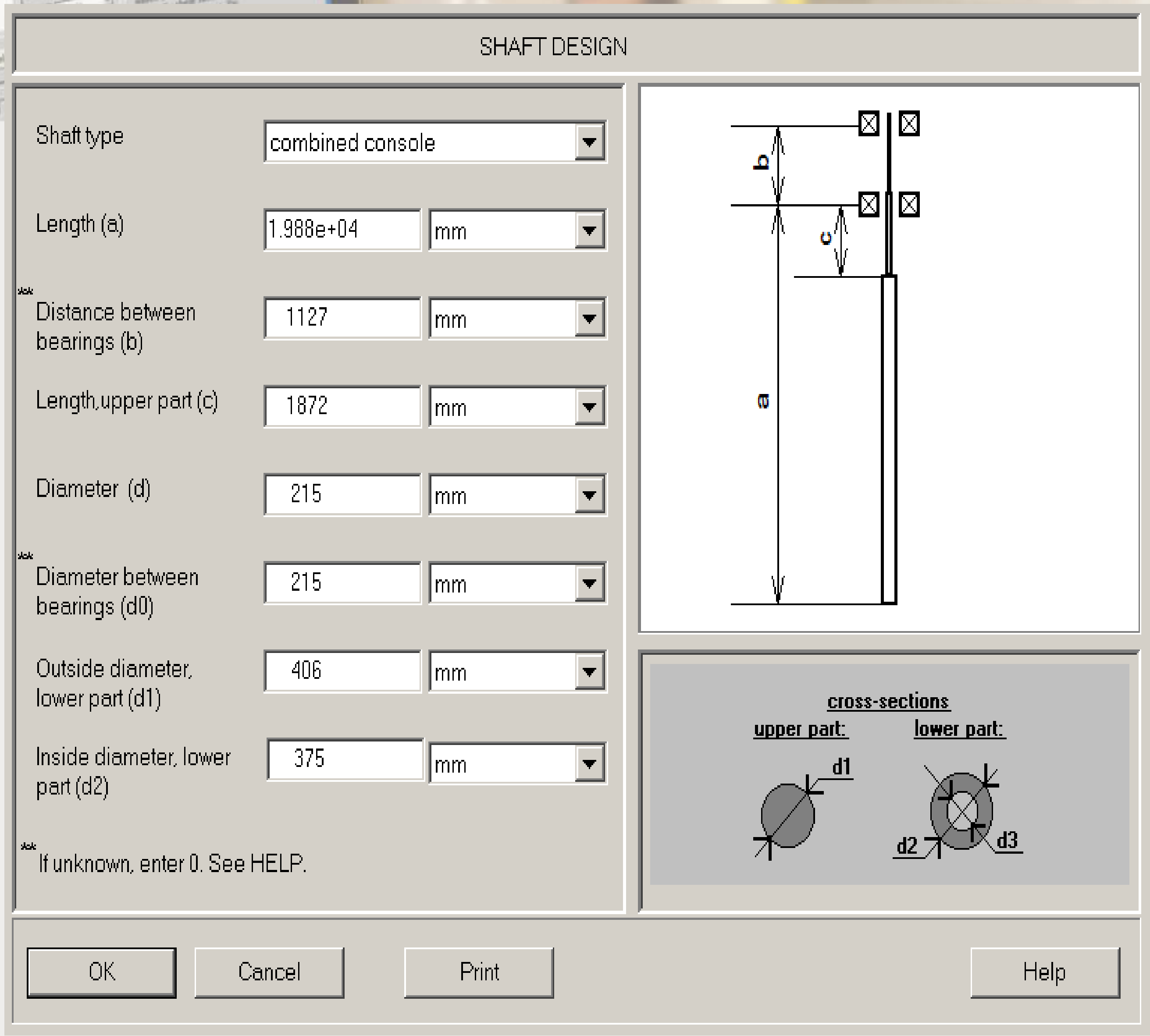
<u>Example 3</u>. Tackling safety problems for *incident/accident investigation.*

The equipment menu of the existing VisiMix® version does not consider *agitators inserted into a draft tube*. Because of this, VisiMix® application is based on the *simplified model* that *differs from the original design by the lack of the draft tube* (see the picture at the right hand side).



VisiMix® application of ISD/IST …. *Example 3* (Cont'd)

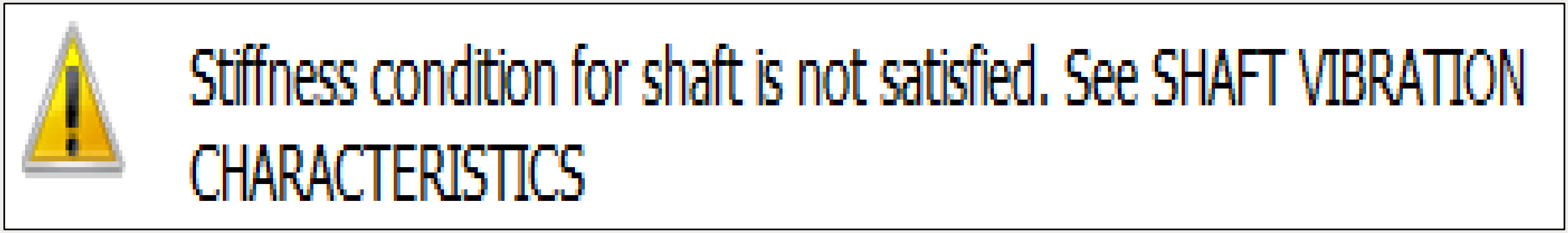
The VisiMix® model of the shaft is presented in the picture below.



VisiMix® application of ISD/IST …. *Example 3* (Cont'd)

Analysis Results. As far as the problem under investigation is connected with the shaft breakage, the VisiMix® submenu **Mechanical calculation of the shaft** was selected for the following study.

This submenu enables to define **Torsion shear** and **Shaft vibration characteristics**. Any of them results in the following warning message.



VisiMix® application of ISD/IST …. *Example 3* (Cont'd)

Checking **Shaft vibration characteristics** results in the following table.

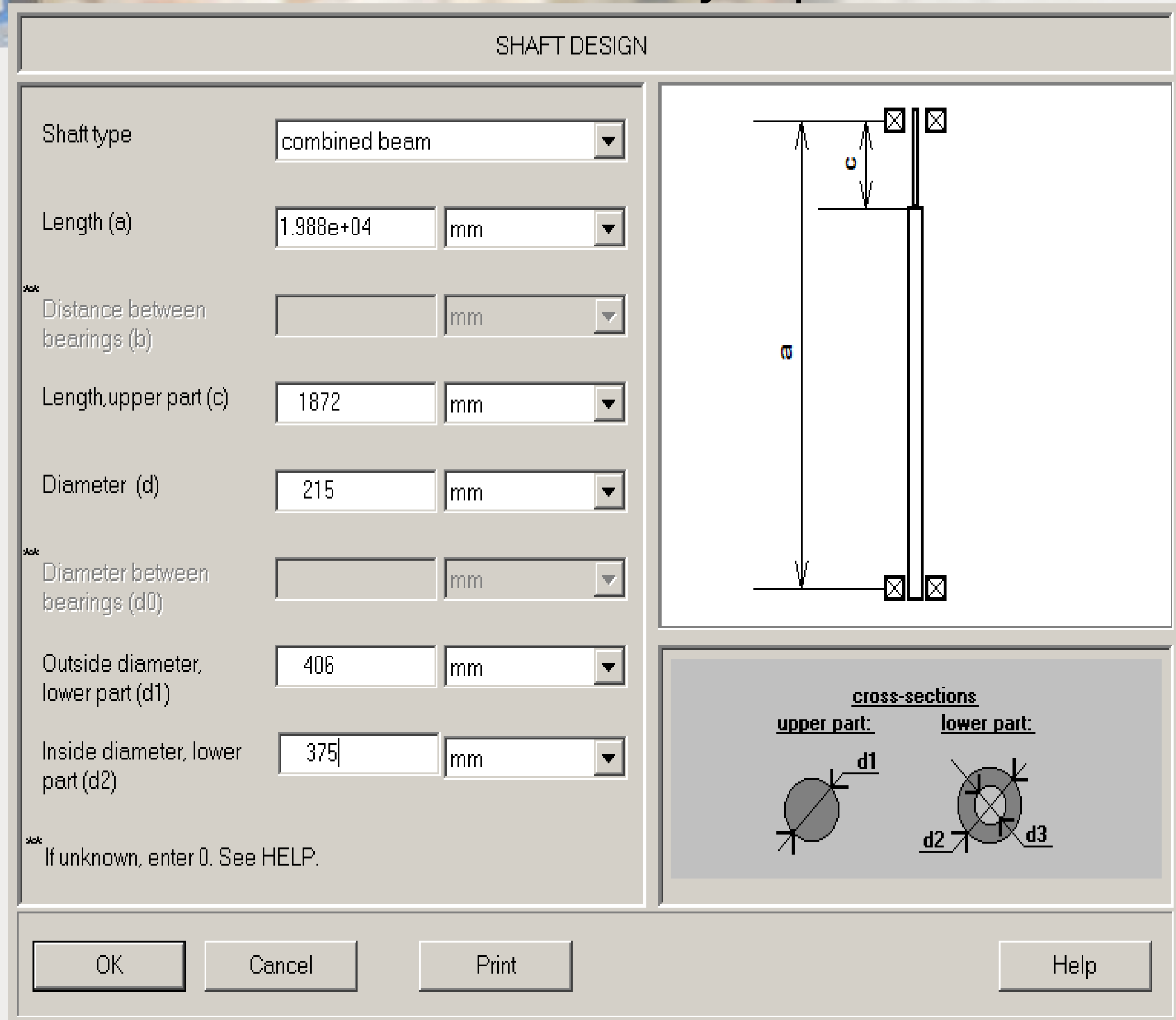
SHAFT VIBRATION CHARACTERISTICS

Parameter name	Units	Value
Critical frequency	1/s	0.309
Rotational frequency	Rps	0.412
Rotational to critical frequency ratio		1.33

The fact, that the *shaft rotational frequency* exceeds it *critical value*, means that after the massive impeller motor was switched on, the *rotation speed increases gradually* from the zero value up to the operation one. This *start regime goes slowly* because of the impeller considerable mechanic inertia and there is a time interval when the shaft rotational frequency becomes close or equal to critical frequency that causes resonance oscillation with possible shaft breakage.

VisiMix® application of ISD/IST …. *Example 3* (Cont’d)

Analysis Results (cont’d). A plausible solution of the above problem is to increase the shaft stiffness. It can be achieved by replacement of of the existing shaft with a mechanical scheme (***combined console***) with a new shaft with another mechanical scheme (***combined beam***) presented below.



VisiMix® application of ISD/IST …. *Example 3* **(Cont'd)**

Analysis Results (cont'd). Checking **Shaft vibration characteristics** results in the following table.

SHAFT VIBRATION CHARACTERISTICS

Parameter name	Units	Value
Critical frequency	1/s	1.55
Rotational frequency	Rps	0.412
Rotational to critical frequency ratio		0.266

The table above conclusively demonstrates that the *rotational frequency of the modified shaft is much below its critical frequency* and thus the modified shaft design does not jeopardize appearance of the resonance oscillations.

Conclusion

An experience of the VisiMix® application enables to conclude the following:

1. Process analysis based on *justified technical calculations and simulations* is a mandatory element of the ISD/IST concept.

Conclusion

2. VisiMix® software has proven its capabilities in simulation various process in stirred tanks/reactors and estimation of their safety ranges because of the following features:

- Provides *adequate* and *complete* simulation of the process with *respect to equipment configuration* based on *reliable models verified in practice*.
- *Detects any deviations of the process course* regarded as factors causing accidents and *estimates influence of these factors influence*.
- It is *user friendly* and allows *quick results*.
- *Combines a calculation tool with an expert system* that analyses initial data and calculation results, and *warns the user whenever the system approaches dangerous operation regimes*.

Conclusion

3. VisiMix® software has demonstrated its efficiency in ISD/IST for:

   (**i**) reactor/process design stage;

   (**ii**) operation stage;

   (**iii**) accident/incident investigation.

<u>Conclusion</u> (Cont’d)

4. A new version of the VisiMix® Turbulent tool will include an *additional submenu specially adapted to ISD/IST problems* and providing an engineer a list of test corresponding to the considered unit operation for revealing of possible sources of troubles.

Thus VisiMix® modeling allows *predict dangerous situations* and *find technical means to mitigate/eliminate the probable risks*.

It means that VisiMix® usage provides stirred reactors a high degree of ***Inherently Safer quality***.

Thank you very much!

Come visit us at our stand A33, Hall 9.1