Theoretical and experimental investigation into the explosive boiling potential of thermally stratified liquid–liquid systems

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Abstract

The occurrence of a rapid phase transition, or so-called explosive boiling, when a cold volatile liquid comes into contact with a hot liquid or hot surface is a potential hazard in industry.

This study was focused on the explosive boiling potential of thermally stratified liquid–liquid systems that result from a runaway reaction. The experimental runs were performed on both a non-reacting and a reacting system.

The experimental results showed that under the analysed conditions, the cold phase was superheated but did not evaporate explosively, as the limits of superheat of the phase were not achieved. The response of the cold phase appeared to be completely controlled by the interface temperature between the hot and the cold phase.

In general, based on the order of magnitude of temperature differences that result from a runaway reaction in a multi-phasic system and the fact that the system is pressurised by its own vapour pressure, the occurrence of explosive boiling under runaway conditions appears unlikely for these type of systems. © 2002 Elsevier Science B.V. All rights reserved.

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1. Introduction

As well-known, runaway reaction or thermal explosion occurs in an exothermic chemical reaction system due to the fact that some chemical reaction rates are very sensitive to the temperature variations connected to a self-heating reaction. For a safe batchwise reaction, wide literature is available on the topics of induction time, under supercritical conditions

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[1] or adiabatic conditions [2], as well as on the critical conditions, according to the simple Semenov model [3,4], or to more complex conditions [2]. Moreover, some particular reaction were deeply studied to determine the critical runaway temperature and the heat transfer coefficient under critical conditions e.g. cumene hydroperoxide reaction [5]. In the chemical industry, there are a number of reactions performed in mixed multi-phasic systems. Examples of these types of reactions are suspension and emulsion polymerisations, or reaction systems in which the reactant and the product are present in an aqueous and an organic phase, respectively (or vice versa). In these systems, accidental loss of agitation might lead to a segregation of the phases and the occurrence of a runaway reaction in one of the phases. The complexity of hazard assessment for these kinds of systems in terms of temperature and pressure excursions can be illustrated by an example on a polymerisation reaction as follows. The scenario that leads to the occurrence of explosive boiling in this case is often considered as the worst case for these kind of systems. Dealing with a suspension polymerisation, the reaction is performed under well-stirred conditions so to obtain the desired product specifications. A malfunctioning of the stirrer will lead to segregation between the aqueous phase and the organic phase in which the exothermic polymerisation reaction proceeds. As a result of reduced heat transfer over the wall of the reactor and a concentration of reactive mass, a runaway might occur leading to a system of a cold water layer with a hot polymer layer (possibly well above 400 °C) on the top of it. A disturbance of the two-layer system (by venting, re-starting the stirrer or a rollover) leads to a flash evaporation of the cold liquid. Subsequently, a fast and unexpected pressure rise occurs as the flashing liquid enhances the mixing of the phases. If significant vaporisation occurs in a short period of time, the process resembles an explosion. Explosive boiling, during which vapour bubbles appear within the boiling droplet, rapidly forming a single growing bubble, occurs when liquid droplets are suddenly and drastically heated by immersion in a hot medium [6].

Although studies on subjects related to the complex nature of the above-mentioned process are described in literature [7–9], little information is available on the potential explosive boiling phenomena related to separation, runaway and vent behaviour of multi-phasic systems. In this study, the prediction of pressure–temperature relations at a sudden mixing of the phases, as well as the characterisation of the effect of flash evaporation on vent requirements and outflow properties were addressed. Apart from the explosive boiling potential that results from a runaway reaction, this study is also relevant for related phenomena like accidental filling of a high temperature reactor with a volatile liquid, application of coolant injection for runaway prevention and equipment failure leading to a sudden contact between phases (heat exchanger or reactor jacket).

2. Theoretical approach

The process of rapid (100–200 ms at atmospheric pressure) phase transition from highly superheated liquid to vapour is known as explosive boiling [10–12]. The superheat limit or homogenous nucleation limit, represents the deepest possible penetration of a liquid into the domain of metastable states. At constant pressure, it is the highest temperature below the critical point that a liquid can sustain, without undergoing a phase transition; at constant temperature, it is the lowest pressure.

Generally speaking, dealing with the type of systems here considered, two ways of reaching the superheat limit can be identified. In the former, at constant pressure, the superheat limit is reached as the temperature exceeds a threshold value. This value depends on the physical properties of the system such as viscosity, density and surface tension and equals the homogeneous nucleation temperature of the liquid. Explosive boiling is more difficult to achieve, as the temperature of the hot phase in contact with the liquid increases well beyond the threshold temperature. Under these conditions, a rapid establishment of film boiling takes place. As a result, a vapour layer is produced, suitable to protect the bulk cold liquid from direct contact with the hot phase. The contact of two phases at different temperatures leads to the heating of a thin film of the cold phase well above its expected boiling temperature. According to [13] the following expression can be used to predict the interface temperature

$$T_{\text{interface}} = \frac{T_{\text{h}}\alpha_{\text{h}} + T_{\text{c}}\alpha_{\text{c}}}{\alpha_{\text{h}} + \alpha_{\text{c}}} \tag{1}$$

in which T_{h} and T_{c} are respectively, the temperatures of the hot liquid and the cold liquid; α_{h} and α_{c} are the "effective thermal diffusivity" of the hot liquid and of the cold liquid, respectively. The "effective thermal diffusivity" is expressed by the following equation:

$$\alpha = \sqrt{C_p \rho \lambda} \tag{2}$$

in which C_p is the specific heat, ρ the mass density of liquid and λ is the thermal conductivity.

It must be remarked that, according to Eq. (1), the interface temperature follows the temperature of the liquid characterised by the highest "effective thermal diffusivity". Explosive boiling occurs if, at the given pressure, the interface temperature exceeds the homogeneous nucleation temperature of the cold phase. A number of small-scale experiments attempted to develop a theory of bubble growth during explosive boiling of a liquid droplet. Under the assumption of vapour temperature equal to the liquid boiling temperature and not equal to the temperature of the superheated liquid, Shusser and Weihs [14] predicted the evaporation rate by the Hertz–Knudsen formula [15], as follows:

$$J = p_{\text{s}} \sqrt{\frac{M}{2\pi R T_{\text{S}}}} \tag{3}$$

where J is the evaporation rate; T_{S} the boiling temperature for the droplet liquid; p_{s} the saturation pressure at T_{S}; M the molar mass of vapour and R is the universal gas constant.

The latter way to reach the superheat limit is, at constant temperature, a fast depressurisation that leads to a pressure far below the saturation pressure of the liquid. In general, due to the effect of pressure on bubble growth rates, explosive boiling is difficult to achieve at high system pressures. At high pressures, vapour bubble growth rates are relatively low and dominated by the rate of heat transfer into the growing bubble. Under these conditions, vapour explosions are difficult to initiate and only rapid (non-explosive) boiling occurs. At low pressures, the bubble growth rates are high and inertially controlled.

An additional complicating factor in the experimental assessment of the phenomenon is the effect of scale. In fact, thermal risk assessment involves both implicit and explicit

use of scale-up principles. At first, routine and rule-based procedures, which contains the scale-up principles implicity are to be used to identify clear cases and cases more sensitive with respect to thermal safety. Progressively extensive effort are then directed to the latter in more explicit scale-up considerations on the basis of thermokinetics models and heat balances [16].

3. Experimental details

3.1. Non-reacting system experiments

In order to study the temperature development and to visually observe the phenomena that might take place at the interface between two thermally stratified liquids, a first series of experimental runs was carried out. The experiments were performed in the so-called constant pressure autoclave (CPA) on a non-real-time system [17]. The two selected non-reacting liquids are water, dyed by chrome-nitrate, and 2,2,4,6,6-pentamethylheptane (isododecane).

The heart of the CPA installation is a glass tube reactor with a diameter of 3.5 cm and a height of 15 cm. The tube is positioned in a containment section of the installation. The gas space of the tube and the containment section are connected via a condenser, so that no pressure difference over the tube is build-up during operation. The headspace of the tube is connected to two large containment vessels, so a nearly constant pressure can be maintained during operations. The installation can be pressurised up to a pressure of 200 bars. A schematic diagram of the installation is reproduced in Fig. 1.

Three chromel–alumel, K-type thermocouples (with a calibrated accuracy equal to 0.5 K) were placed in the tube to measure the water, interface and isododecane temperature. The location height of the thermocouples were 2.8, 6 and 10.8 cm from the bottom. In the given set-up, the temperature of the water and isododecane phase can be modified by adjusting the power to the heater at the bottom of the CPA or the power to a heating spiral around and within the top half of the test tube.
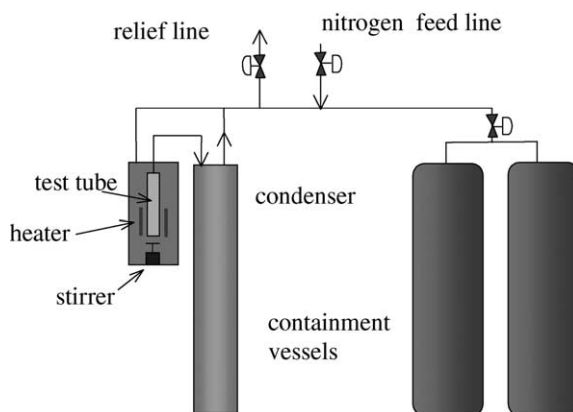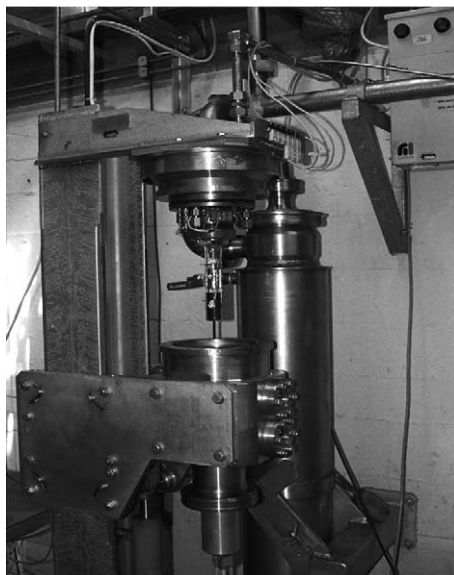

Fig. 1. Schematic diagram of the CPA.

Fig. 2. Picture of the CPA.

Upon filling the test tube with a pre-defined amount of water and isododecane, the tube is placed in the CPA, depicted in Fig. 2, which is closed and pressurised. By means of a video camera (Panasonic CCD 50 frames/s 1/16000 exposure time) localised in front of the autoclave, it was possible to record the process occurring at the interface. The pressure and the temperatures are controlled from the remote control system. The recorded images and the measured values of temperature and pressure are recorded by a computer system, for subsequent processing and evaluation.

In order to evaluate heat exchange between the phases and the vapour pressure trend, preliminary experimental runs with a non-reacting system were also performed by means of the controlled runaway and vent monitor (CRVM) described in detail in the following paragraph. Two preliminary series of experiments were carried out with a mixture of water and 2,2,4,6,6-pentamethilheptane at 50% (w/w). The reactor was filled with a standard amount (400 g) of the mixture and was subsequently heated up to a temperature of 483 K. At a liquid temperature of 483 K, the relief line was opened. In the first series of experiments, the system was perfectly mixed by the stirrer set at 900 rpm; in the second series, a complete separation of the organic phase from the aqueous one was obtained, maintaining the stirrer off.

3.2. Reacting system experiments

Upon the preliminary experiments with a non-reacting system, a two-phasic reacting system was selected, for experiments on a 1-l scale. To this purpose, the decomposition reaction of an organic peroxide (dilauroyl peroxide) in a water–isododecane mixture was

investigated. Apart from the studied phenomena in the preliminary experiments, the experimental runs on the 1-l scale were also performed to study the effect of flash evaporation on vent requirements and outflow properties. The study on the reacting system included, as well, the study of the system behaviour upon pressure release.

The experiments were performed in the so-called CRVM [17]. The CRVM was designed and developed ad hoc, so to characterise the thermal properties and vent behaviour of high energetic chemicals. The experimental set-up consisted of a 1.1-l reactor (height over diameter equals 1.4), capable of operating up to a pressure of 250 bars at 623 K, connected to a process control and data acquisition system. The reactor is equipped with a flat-blade turbine impeller stirrer, a piezoresistive pressure transducer (natural frequency 1600 kHz, calibrated to an accuracy of about 2% of the measured value), multiple chromel–alumel, K-type thermocouples (four internal and two mounted in the vessel wall), a 0.5-in. bursting disk, electrodes for the ignition of the head space or for the use of an internal heater, two fill/relief lines (diameters of 3.7 and 9 mm) and a bottom drain. The bottom drain can be used for either emptying the reactor, or for operating the reactor as a continuously stirred tank reactor.

Two helical heaters placed at the top and bottom sections heat the reactor vessel. Both heaters are independently controlled by programmable process controllers, which, in turn, are operated on the basis of a dynamic temperature programme selected on the process control and data acquisition system. As a result, the reactor can be operated with an imposed temperature programme (constant power, constant wall temperature, constant temperature increase) or pseudo-adiabatically. In the pseudo-adiabatic mode, the wall temperature, measured by the thermocouples positioned within the vessel wall, is kept equal to the temperature of a selected internal thermocouple (within 0.5 K) by controlling the amount of heat supplied to the vessel. The maximum temperature rise rate of the tested substance that can be compensated in the pseudo-adiabatic mode, equals approximately 10 K min^{-1}. A schematic diagram of the experimental set-up is reported in Fig. 3(a) and a picture of the reactor is presented in Fig. 3(b). Six experiments were carried out, with three different concentrations of peroxide in the organic phase, corresponding to 50, 75 and 100% all in wt.%. Each concentration was tested with and without back-pressure. In the experiments performed with back-pressure, the reactor was connected, via the 3.65 mm diameter relief line, to the gas containment section of the CPA.

The experiments were performed according to the procedure described in the following. Firstly, the reactor was filled with the pre-defined amount of water (400 g). Subsequently, the reactor was heated to a temperature of approximately 333 K. At the same time, the mixture of isododecane and peroxide was prepared. To easily dissolve the peroxide in isododecane, the dilauroyl peroxide was melted first and then added to the solvent (the melting point of the peroxide equals 329 K). To prevent an early decomposition of the peroxide, the temperature was carefully controlled below 333–338 K. The prepared peroxide mixture (with a total mass of 320 g) was introduced in the reactor and mixed with the water, having set the speed of the stirrer at 900 rpm.

The top heater of the reactor was switched to a 100% power. The relatively fast heat-up of the top of the reactor was done to reduce heat loss during the later stage of the runaway. The bottom heater of the reactor was switched off or lower than 40% of power at the beginning. At a liquid temperature of 363 K, the stirrer was switched off, upon which a separation of
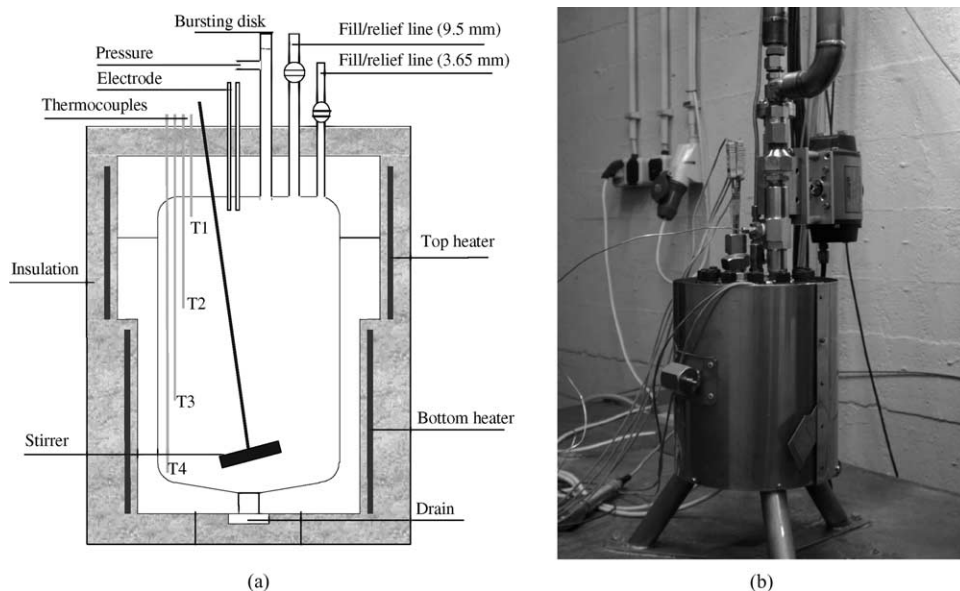
Fig. 3. (a) Schematic diagram of the controlled runaway and vent monitor; (b) photograph of the reactor.

the phases occurs. After the separation, the temperature of the gas phase was measured by thermocouple T_1, the organic liquid temperature by T_2 and T_3 (with T_3 close to the interface between the organic and the water phase) and the water temperature by thermocouple T_4.

Due to the high temperature of the organic phase, and the ongoing heat-up of the reactor, a runaway occurs in the organic phase upon the phase separation. Pressure and temperature of the organic liquid increase rapidly due to the runaway. When pressure reaches its maximum, the 9.5 mm with a restriction with a diameter of 1.8 mm is opened. In the experiments with back-pressure, the pressure is relieved via the 3.65 mm valve in the pressurised gas containment section of the CPA.

4. Experimental results

4.1. Non-reacting system

Four experiments were performed at increasing pressures, namely 4.5, 9, 24 and 40 bars. For each experiment, the theoretical interface temperature was calculated on the basis of heat transport between the hot and cold layer, according to Eq. (1). The temperature curves measured in the experiment carried out at a pressure of 4.5 bars are presented in Fig. 4. During this experimental run, a few minutes after the start of the experiment, boiling took place at the interface between the liquids. The boiling occurred as follows: first, a large bubble is produced at the interface. When the bubble was expelled from the interface, another bubble was formed. At the beginning, this phenomenon took place at a low speed, but after
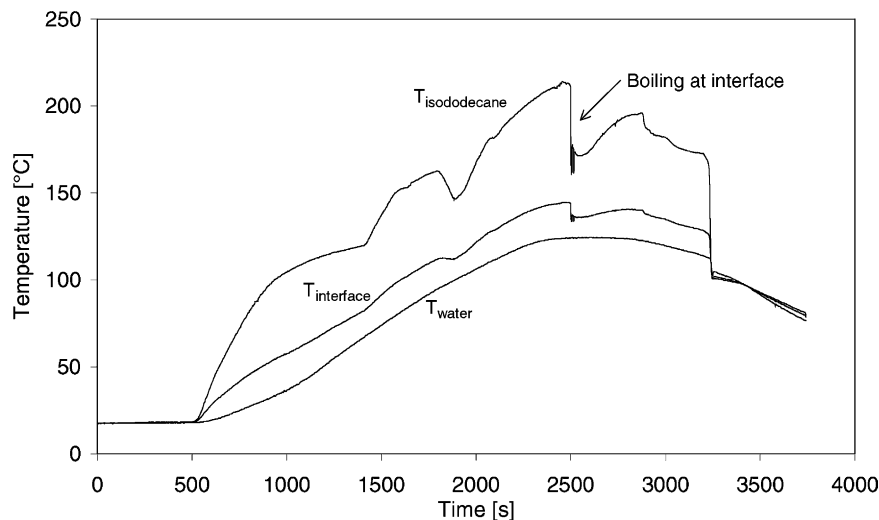
Fig. 4. Experimental run with water and 2,2,4,6,6-pentamethylheptane (50% w/w) at a constant pressure of 4.5 bars.

a while, it occurred at a higher speed producing tiny bubbles and fine-scale fragmentation at the interface.

Several replicated experiments were performed to determine correctly the onset of the phenomenon. At temperature difference between the liquids of about 90 K (2500 s), a fast boiling process was observed at the interface between the liquids. It must be observed that the irregularities in the temperature curve prior to this boiling effect were caused by adjustments in the power supply to the heating spiral. The fast boiling occurred in connection with a fast decrease of the isododecane temperature. This decrease of the isododecane phase was caused by the evaporation of water. The evaporation of water, as well as the heat transport by water vapour, extracted heat from the isododecane phase. The measured temperature curve in the water phase clearly shows that the volatile boiling was in fact restricted to the very top of this phase. The occurrence of the fast boiling process at the given conditions can be explained by a consideration of the vapour pressure at the top of the water phase and the total pressure of the system. The vapour pressure at the top of the water phase was calculated from the temperature at the liquid interface, which, in turn, was calculated from the measured water and isododecane temperatures. The calculated interface temperature at the fast boiling process was 425 K, which corresponds to a vapour pressure at the top of the water phase exceeding the initially imposed pressure on the system. Hence, boiling occurs.

The experiment carried out at 9 bars only showed boiling at the top of the aqueous phase. The experiments carried out at 24 and 40 bars showed no boiling, even upon pressure relief. The absence of boiling was caused by the fact that water temperature was still below its atmospheric boiling temperature. The results of the experiment at 40 bar are shown in Fig. 5 and evidence fairly good agreement between the calculated and the measured temperature at the liquid interface. Furthermore, the figure clearly shows that the interface temperature
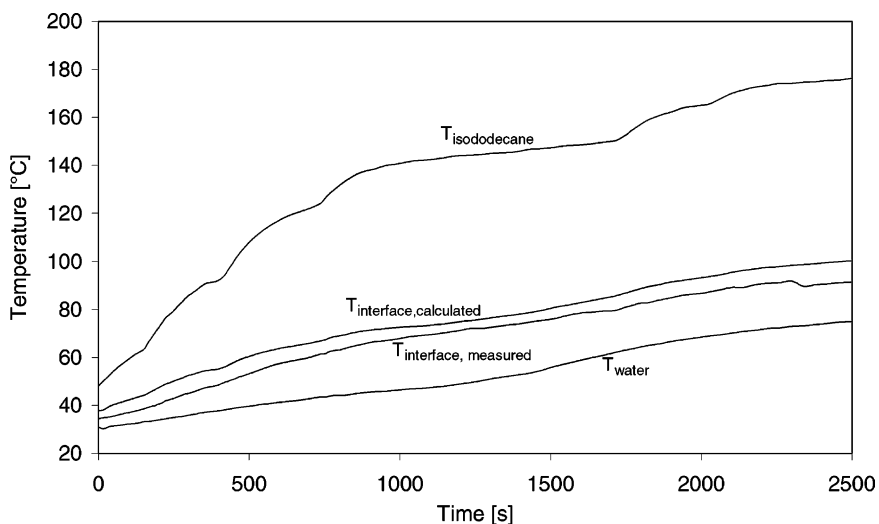
Fig. 5. Experimental run with water and 2,2,4,6,6-pentamethylheptane (50% w/w) at a constant pressure of 40 bars.

follows the temperature of the liquid that has the highest thermal diffusion. Based on the experimental findings, it can be concluded that Eq. (1) provides a good estimation of the interface temperature. Apart from an experimental verification of this equation, the preliminary experiments contributed considerably to the understanding of the interfacial boiling phenomena and the pressure effect on these phenomena.

Main results of the experimental runs carried out by CRVM are reproduced in Figs. 6 and 7, respectively for perfectly mixed and completely stratified conditions. The exact position of the four thermocouples is also reported in the above-mentioned figures. During the heating phase, it was verified a common trend, so that the simple Raoult's law [18] can approximate the vapour pressure trend. On the contrary, the relief trend modelling appears highly dependent on stirring conditions. In fact, dealing with the stratified system (Fig. 7), the trend during the whole relief period, starting from a pressure of nearly 18 bars, can be described by a flash evaporation of the separate two components. In the completely mixed system, an adiabatic flash evaporation of an homogeneous mixture describes the behaviour in the first phase of the relief, down to a pressure of 8 bars ca., while Raoult's law approximate the trend in the final relief period.

4.2. Reacting system

The key values of the experimental runs are summarised in Table 1.

In general, by comparing the experiments carried out at different peroxide concentration, same common features can be outlined. The experiments performed with a peroxide concentration of 75% (w/w) are discussed thoroughly in the following. In principle, each experiment consists of two steps. The former is the stage in which the phase separation and runaway reaction takes place. The latter step is the actual pressure relief,
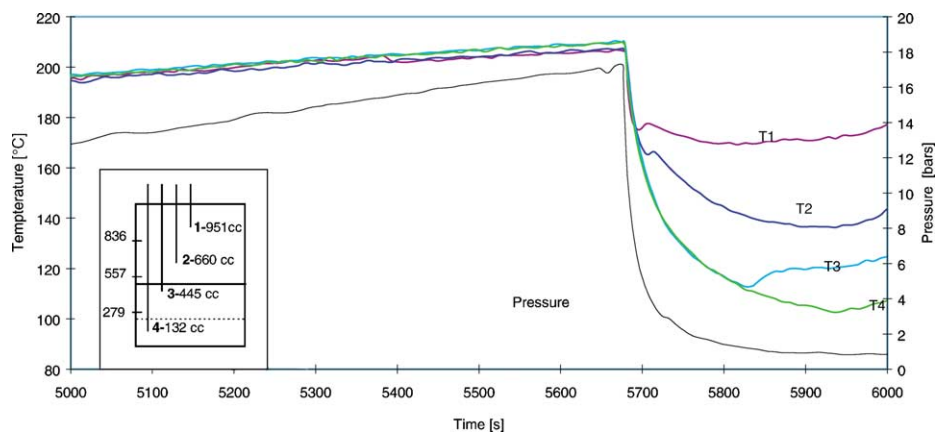
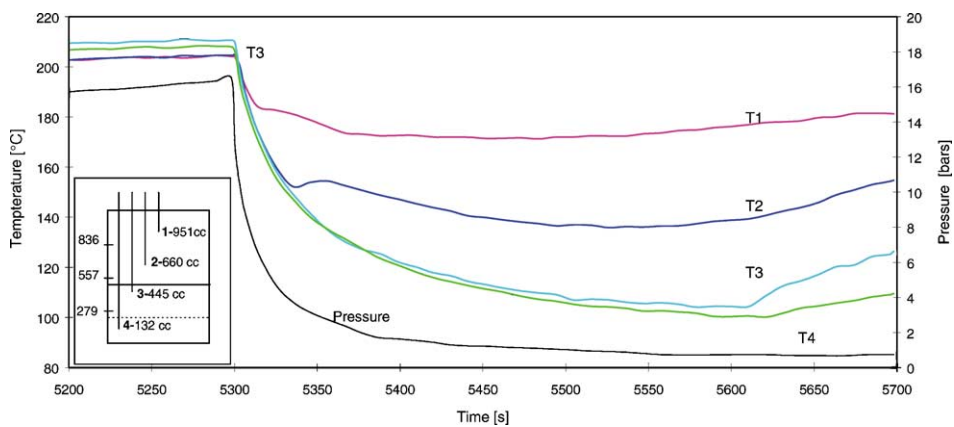Fig. 6. CRVM experimental run with water and 2,2,4,6,6-pentamethylheptane (50% w/w), under perfectly mixed conditions.

Fig. 7. CRVM experimental run with water and 2,2,4,6,6-pentamethylheptane (50% w/w), under completely stratified conditions.

Table 1
Key results of the experimental runs carried out with a reacting system (water and peroxide)

Peroxide concentration in organic phase (% w/w)	50	75	100
Onset temperature of the runaway reaction (K)	343	343	354
Maximum pressure (bars)	40	62	94
Maximum temperature of the organic phase (K)	478	513	553
Temperature difference at pressure relief (K)	100	140	200

either to the back-pressure of the gas containment system of the CPA, or to the ambient pressure.

Fig. 10 shows the results of the experiment with a peroxide concentration of 75% (w/w). In the initial stage of the experiment, when the phases are still thoroughly mixed, a decomposition reaction of the peroxide occurs. The temperatures within the system are equal, up to the moment at which the stirrer is switched off (at about 363 K). Without mixing, a fast separation of the two-phase mixture takes place, resulting in an aqueous phase with an organic phase on top of it.

The runaway reaction in the organic phase clearly accelerates upon the phase separation due to the concentration of the reactive mass. Fig. 8 shows that, due to stratification within the organic phase and the fact that T_3 is measured close to the water phase, the runaway starts at the top of the organic liquid. Upon reaching its maximum value, the temperature rise in the top layer of the organic phase stops, due to reactant depletion. At this stage, the runaway in the layer directly below the top layer of the liquid is still proceeding. As a result, the temperature of this layer approaches the temperature of the top layer. This process proceeds as time goes on and gives account of the correlation between T_2, T_3 and the pressure increase. It is noteworthy observing that the temperature measured in the water phase (T_4) does not correspond to the runaway condition.

After reaching its maximum, the pressure begins to decrease slowly, due to heat loss from the gas phase to the top of the vessel. Just at the moment the valve is opened, a steep drop in pressure to the back-pressure in the CPA system is observed. During the depressurisation, T_1 increases rapidly, whereas the temperatures measured as T_2 and T_3 reduce. This reduction in temperature is caused by the evaporation taking place at the liquid interface. The produced vapour mixes the organic phase and contributes to the heat removal
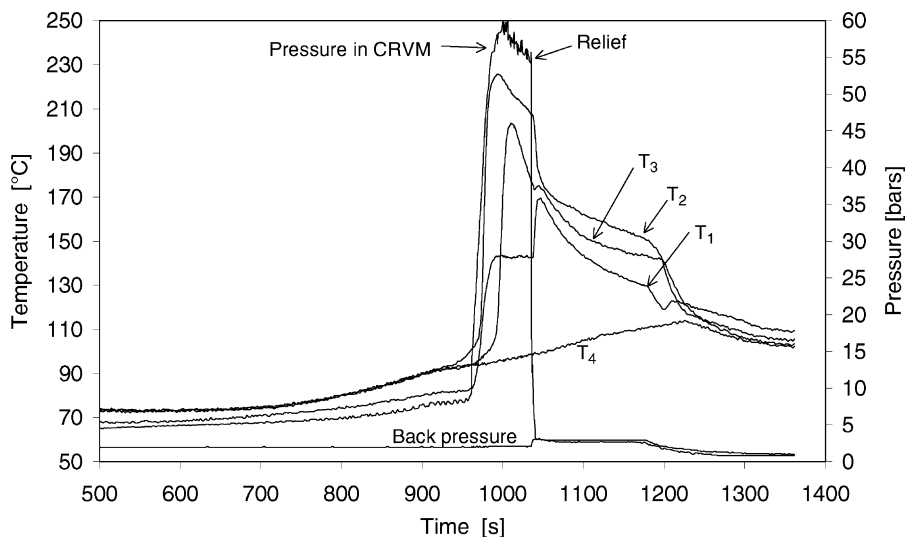

Fig. 8. CRVM experimental run with a mixture of peroxide and water (75% w/w), with 2.3 bars back-pressure during the relief phase.
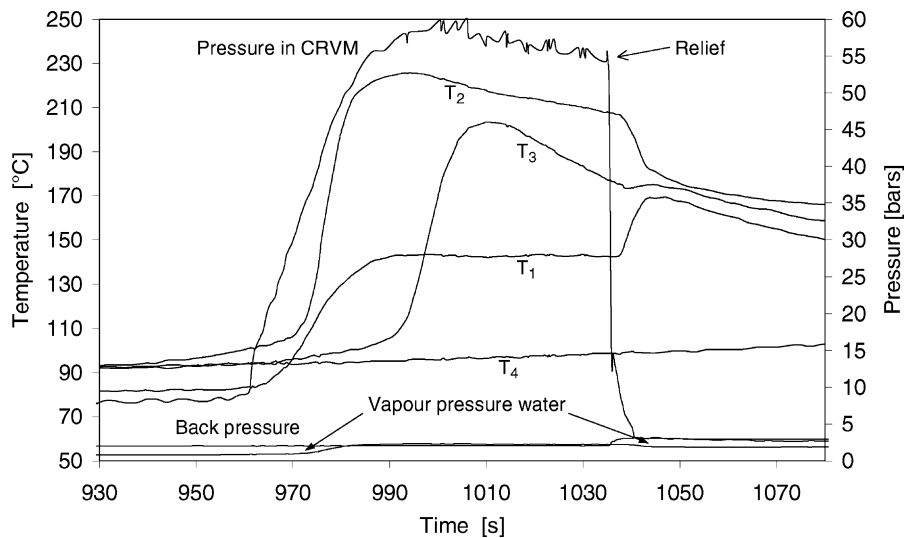
Fig. 9. Detail of the relief period with a back-pressure of 2.3 bars with a mixture of peroxide and water (75% w/w).

by its heat capacity. It must be noticed that an initial temperature drop of T_2 is measured, due to the evaporation of light (volatile) reaction products from the organic phase. The vent process of the experiment is presented in more detail in Fig. 9, referring to the results of the experiment with an imposed back-pressure.
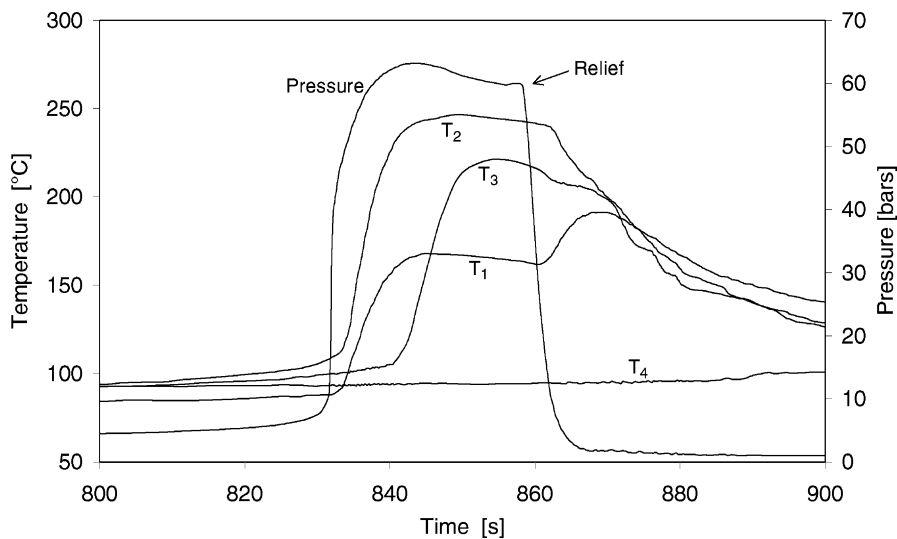

Fig. 10. CRVM experimental run with a mixture of peroxide and water (75% w/w), with no back-pressure during the relief phase.
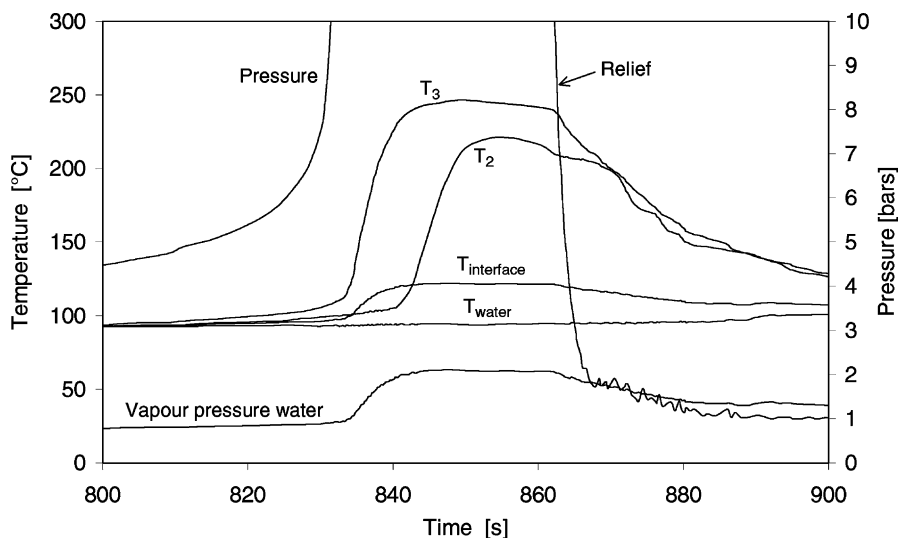
Fig. 11. Detail of the relief period with no back-pressure with a mixture of peroxide and water (75% w/w).

The results of the vent process to ambient pressure are shown in Fig. 10. From these results, it can be observed that the part of the experiment prior to the pressure relief is equivalent to the counterpart shown in Fig. 8. However, in contrast with the experiment with back-pressure, a flash evaporation of water is observed at the end of the relief period. A detail of the vent process during the experimental run performed with no back-pressure is depicted in Fig. 11.

Both the severity of the process of flash evaporation and the pressure at which it starts increase as the peroxide concentration increases. A high degree of correlation ($P < 0.005$) between the evaporation process and the temperature at the interface T_2 was verified.

5. Discussion

According to the theoretical approach and the experimental results (namely temperature of the water phase and the organic phase), the theoretical interface temperature $T_{\text{interface}}$ and the vapour pressure of water at the interface were calculated for each experiment. The calculated curves are also depicted in Figs. 9 and 11. A comparison between the experimental curves, obtained with and without back-pressure, and the theoretical information resulting from the calculated vapour pressure curves, highlights that the back-pressure prevents the flash evaporation of water from the interface. The main conclusion that can be drawn from the above is that the process of flash evaporation does only occur when the vapour pressure of water at the interface exceeds the overall pressure of the system.

None of the experiments showed explosive boiling, as the criteria for a rapid phase transition or explosive boiling were not met. This was especially verified for the criterion on the limit of superheat of the cold liquid at the given temperature differences between the

two liquids. It should be noted that, in general for vapour systems, high levels of superheat are not easily reached as the system pressurises itself. Furthermore, the pressure drop upon initiation of a vent process remains small.

It is clear that dealing with thermal hazard assessment, scale-up considerations, from the laboratory to the real plant, play an important role. It should be noted that, for large-scale events, the liquid must be pre-fragmented at the inception of explosive boiling. Whether or not this condition is met on the large-scale (by the initial flash evaporation) is hard to predict from small-scale experiments. In general, with respect to the boundary condition on pre-fragmentation, small-scale experiments appear to be conservative. In fact, on the large-scale, runaway reactions will not usually proceed homogeneously in one phase. As a consequence, it is foreseeable that the results of a runaway reaction, in terms of temperature and pressure build-up of the investigated system, are smaller on a large-scale than the ones obtained in the tested small-scale, under well-stirred and well-controlled conditions.

6. Conclusions

Experiments with a non-reacting system have shown that water was superheated but did not evaporate explosively, as the threshold limits of water superheat were not achieved. There was only heat transfer by nucleate boiling. Further it was evidenced that the temperature at the interface between the hot and the cold phase can be calculated on the basis of the bulk temperatures of these phases. The experimental results learned that the interface temperature, and in turn, the vapour pressure that results from the interface temperature, are the determining parameters controlling the response of the cold phase.

The maximum vapour pressure exerted by the cold phase can be estimated on the basis of the interface temperature. A vapour pressure higher than the ambient pressure results in a flash evaporation of liquid, from the top of the cold phase (at relatively low levels of superheat), during venting. A vapour pressure lower than the ambient pressure results in evaporation of the cold liquid, up to the level at which thermodynamic equilibrium with the gas phase is attained. The vapour production rate from the cold phase during a relief process is controlled by the given vapour pressure and the volumetric relief rate.

The occurrence of a vapour explosion on a large-scale can only take place when the level of superheat approaches the level at which homogeneous nucleation takes place and when fragmentation of the cold liquid takes place. Based on the order of magnitude of temperature differences that result from a runaway reaction in a multi-phasic system and the fact that the system is pressurised by its own vapour pressure, it appears to be unlikely that high levels of superheat are reached. Hence, it can be stated that the occurrence of a vapour explosion under runaway conditions is, in general, unlikely. The results appear to be representative also for larger scale, especially considering that the constraint for runaway on a large-scale is pre-fragmentation of the phases. This constrain was certainly met during the experimental runs carried out on the laboratory scale but might not be met on the large-scale.

In any case, the results are very sensitive to the values of the fluid properties; therefore particular care should be taken when evaluating these. Due to its dependence upon many factors, evaluation of the conditions under which explosive boiling can take place is a complex problem that should be approached with care and requires in-depth investigation.

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