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Temperature Measurement in Heat Transfer

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1. Introduction

Accurate measurement of temperature plays a vital role in many fields of engineering and scientific practice not the least for energy and environmental issues. In recent years non-invasive techniques such as Liquid Crystal Thermography (LCT) and Infrared Thermography (IRT) have been prevailing on many other regular methods like bimetallic thermometers and thermocouples. Both LCT and IRT are very useful to get full field properties, i.e. obtaining temperature data of a full surface, from every single point but not average of that area. They allow the user to *map* the temperature of a specific surface regardless of its shape. These techniques have been used many times in the field of heat transfer and more specifically to determine the heat transfer coefficients of different shapes. Other common applications are the temperature measurements of electronic devices, which is closely related to their overall performance, and body parts such as breasts, helpful to diagnose breast cancer (Davison, et al. 1972), and feet plants, helpful to diagnose diabetic foot ulcers (Stess, et al. 1986).

Next, we will briefly expose the conceptual basis and main characteristics of LCT and IRT techniques.

Liquid Crystal Thermography

This technique is based on liquid crystals, a material with a state of matter between solid and liquid phases and thereby sharing properties of both liquids and solids. Depending on its molecular structure the phase behaviour of liquid crystals can be divided into three main groups, named as nematic, smectic and cholesteric. LCT is based on the last group of these three, the cholesteric liquid crystals also known as thermochromic liquid crystals (TLCs). These are used as temperature sensors because they change their surface colour depending on its temperature. At a particular temperature TLCs reflect the incident light within a specific band of wavelengths. This selective reflection is based on the molecular structure and it's a complex phenomena set by fiddly formulas. However, no further review about this topic will be developed in the present report.

When illuminated by white light and at temperatures below the TLC's event temperature a liquid crystal is at a solid state in it will appear transparent (colourless) or black if it is sprayed on a black background. As temperature rises the TLC colour varies from red to blue, going through orange, yellow and green. When TLC reaches the clearing point it enters the pure liquid state and becomes colourless again. This process is reversible with cooling. The main fact that is worth bearing in mind is that the colour of the TLC can be related to a specific temperature by a calibration process. Further information about this process will be expounded in this report.

Information about LCT principles and fundamentals was obtained from many different sources, some of them were (Childs 2002), (Gao 2002) and (Sundén 2011).

Infrared Thermography

Infrared Radiation Thermography (IRT) is one of the examples of infrared imaging science. Thermographic cameras detect radiation in the infrared range of the electromagnetic spectrum (roughly 9,000 – 14,000 nanometres or 9 – 14 μm) and produce images of that radiation, called thermograms. Nowadays infrared thermometers

are widely available and measure the thermal radiation emitted by a body due to its temperature. Since infrared radiation is emitted by all objects above absolute zero according to the black body radiation law, thermography makes it possible to see one's environment with or without visible illumination. The amount of radiation emitted by an object increases with temperature. Therefore, thermography allows one to see temperature variations in time as well as in space.

Experimental work

This report presents experimental work carried out using the techniques described above, both LCT and IRT. We began by reading some literature about these techniques. Some papers with experimental studies that used LCT and/or IRT as measurement tools and some other with explanations about the fundamentals of these techniques.

After the literature review, a calibration process of the LCT was done in order to associate temperatures with their corresponding colour. The calibration of LCT associates each temperature to a hue value and it's essential when using liquid crystals to get full field temperatures. Due to the curves temperature - hue value obtained in the calibration not only a qualitative but also a quantitative analysis of the temperatures of a specific surface can be performed.

In order to calibrate the liquid crystal available at the laboratory a set of tools was used. It included, among others that will be described further in this report, a CCD camera and an aluminium plate with liquid crystal sprayed on it. Quite briefly, the calibration process consisted of a heating system that was used to increase the temperature of the liquid crystal while some thermocouples, evenly fixed on the aluminium plate, measured the surface temperature and a CCD camera took pictures of the LCT surface. Matlab software was then used to process all the data gathered. The scripts used to do so can be found in the back-up documents of the present report. One should bear in mind that the main purpose of the calibration is to get a *temperature - hue value* curve for the liquid crystal.

Secondly, once the calibration of the LCT was done, an experiment was carried out. It consisted of a plexiglass plate heated by a resistor and an IR camera as well as a liquid crystal sheet used to measure its temperature. The plate was heated until it reached the steady state and then its surface temperature was measured using both the IR camera and the liquid crystal sprayed on top of it. Again a CCD camera was used to take a picture of the liquid crystal in order to get the hue value for each pixel. The IR camera gives the temperature of the surface directly and no further data process is required whereas the CCD camera gets the hue value of the liquid crystal. Therefore, the data stored with the CCD camera needs to be processed so the temperature can be shown. To do so the function/curve obtained in the calibration is applied to each pixel using the Matlab software. Then the temperatures were used to obtain the heat transfer coefficients and the Nusselt number of the different areas of the surface. The results obtained as well as further and deeper explanations about the procedures and the experimental work carried out will be expounded in this report.

What is in the report?

After this introductory section, in this report the viewer will find, firstly, an introduction to the theoretical basis of liquid crystal thermography as well as an explanation of the

calibration process carried out in the laboratory. Secondly, a statement about the experiment developed to measure heat transfer coefficients and Nusselt numbers of a specific surface will be made. In this phase an overall description of the infrared thermography (IRT) will also be shown. In addition all the Matlab scripts used to process the data gathered can be found in the back up documents of this report.

Finally the report includes a detailed analysis of the results obtained in both calibration and experimental work processes. Within this section the viewer will be able to find a discussion about main advantages and drawbacks of each technique (LCT and IRT) as well as explanations about error analyses among other issues.

The main purpose of this project is to approach two different temperature measurement techniques that have been being applied in the recent years in many several fields of technology. Based on both the experimental and theoretical work done this project should allow us to get familiar with these techniques.

2. Liquid crystal Calibration

As stated before in the introductory section of the present report Liquid Crystal Thermography requires a calibration process in order to relate each colour to a single temperature value. The main purpose is to get an accurate temperature – hue value (colour) curve so it can be applied later to obtain the temperature of a surface. This process is very important for high accuracy and resolution in temperature measurements. As seen before, LC colour response to different temperatures depend on some different factors such as light's degree of incidence or environment reflections.

The calibration process of a liquid crystal can be done either in-situ, i.e. in the same place and under the same optical and environmental conditions that the final thermal measurements will require, or in a separate system. In this case the calibration was made in a separate system. This kind of calibrations, though, might result in some problems. It can be difficult to set exactly the same conditions for the calibration that the LC will experience in the final experiment. Nevertheless, an in-situ calibration may require more thermocouples and it could be hard to fix them properly. These facts could lead the in-situ calibration to be more time consuming. Hence, it is usually easier, faster and more practical to calibrate a liquid crystal in a separate plate where environmental conditions and the tools required can be set more accurately.

Some years ago the calibration of liquid crystals was usually done by human visualization. However, in the recent years many technologies have appeared and evolved and contributed to make it much more precise. One of these techniques is the use of CCD cameras, which can be used to take pictures of the liquid crystal, followed by a digital processing to get full-field distributions of properties such as RGB and HUS data.

In the calibration process carried out in this course a wooden black box with a front window made of plexiglass and some holes on the backside was used. The front window allowed changing and manipulating the plate while the backside holes were made to evacuate the hot air coming from inside. The plate with the liquid crystal was fixed inside the box and was heated with a fan and a heater plugged into the box

through a small hole on the bottom. Two fluorescent lamps were mounted vertically right in front of the plexiglass window to illuminate the plate. The CCD camera was fixed with a tripod in front of the plexiglass window and perpendicular to the centre of the plate. Its height was adjustable. The CCD camera was then plugged into the computer and specific software helped to process the images and select the specific area of interest.

The plate was made of aluminium, since its thermal conduction coefficient is high enough to ensure that the temperature on its backside, where thermocouples were fixed at, was the same as the temperature in the front side, where the LC was fixed.

Five thermocouples were used in total to measure the surface temperature of the plate; they were evenly fixed on the plate using some adhesive tape. The other endpoint was submerged into an ice bath in order to set the reference temperature. A digital multimeter was used to read the voltage difference measured by each thermocouple. Afterwards, the following equation gave the temperature value for each voltage.

$$T[^\circ\text{C}] = 24,467 \cdot U[\text{mV}] + 0,2816 \quad (2.1)$$

The power supplied to the heater was adjusted with a variable transformer. It allowed varying the voltage supplied. The event temperature of the LC was reached by supplying 135V to the heater. At this point the red colour appeared for the first time.

The calibration was completed within two days. Each day data were gathered from red colour to blue, i.e. varying the voltage from 135V to 165V. In total 23 points (Temperature, Hue value) were obtained. The temperature for each point was obtained by averaging the 5 measures captured with the five different thermocouples. The hue value was obtained averaging the hue value of each pixel from the images taken with the CCD camera. Matlab was used to process all this information and data, scripts can be found in the back-up documents.

At the beginning of the calibration the power supply was adjusted at 135V where red colour started to appear. The power supplied to the heater was then increased by 2,5V approximately in every run. After waiting for 20 minutes, in each run, the steady state was reached and all the data regarding temperature and colour were gathered, i.e. temperatures of thermocouples were read and an image of the plate was taken with the CCD camera.

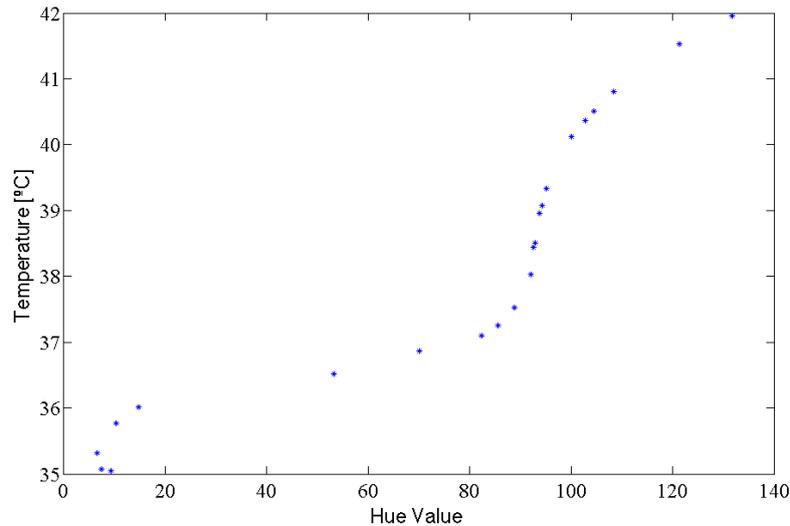


Figure 1: Points Temperature-Hue obtained in the calibration process

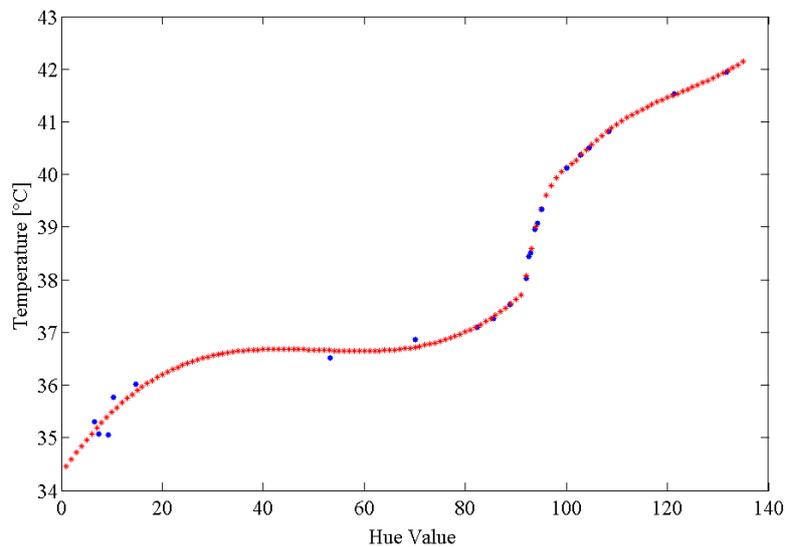


Figure 2: Calibration curve Temperature versus Hue value adjusted to all the data gathered.

Figure 1 shows all the points obtained during the calibration process of the liquid crystal. More points were obtained in the 37°C – 40°C range than in the other sections due to the fact that this segment is considered to be the most sensitive, i.e. little variation of the hue value is related to a big variation of the surface temperature.

Figure 2 represents the adjusted Temperature-Hue curve obtained with Matlab. The full range of the hue value was divided into three different zones: 0-80, 80-100 and 100-135 in order to adjust the curve more accurately. Three-degree polynomial functions join the entire number of points in each section.

As figure 1 shows the first section, with a hue value ranging from 0 to 80, is the most inaccurate since there are a couple of points whose coordinates do not fit the curve very well.

As figure 2 shows those areas or sections where curves are less accurate are the joints between functions 1 and 2 and functions 2 and 3. No better adjustment could be achieved using polynomial functions.

Functions obtained for the three different segments were:

$$T(x) = \begin{cases} 1,8 \cdot 10^{-5} \cdot x^3 - 0,0029 \cdot x^2 + 0,1452 \cdot x + 34,3062, & 0 < x \leq 92 \\ 0,00202 \cdot x^3 - 0,6140 \cdot x^2 + 62,1425 \cdot x - 2,0586 \cdot 10^3, & 92 < x \leq 102 \\ 5,1 \cdot 10^{-5} \cdot x^3 - 0,0190 \cdot x^2 + 2,3722 \cdot x - 58,8851, & x > 102 \end{cases} \quad (2.2)$$

The variable x represents the hue value and the temperature results in °C.

3. Experimental Work: IRT & LCT

When the temperature – hue value curve of liquid crystal has been obtained, it is possible to get temperature distribution of a surface by using liquid crystal. This part of experiment is mainly focused on comparing the accuracy of temperature measurement of Infrared Radiation Thermography (IRT) and Liquid Crystal Thermography (LCT).

As said before, the experimental work carried out with IRT and LCT consisted of a plexiglass plate heated by a resistor and a thin layer of liquid crystal sprayed on the central area of the plate. The exact dimensions of the plate were 0,316 *m* in width and 0,246 *m* in height. In order to prevent the influence of the contact resistance used to heat the plate, the area of study avoids the two ends of the resistor and takes the central area where the temperature is more uniform. The plate was heated until it reached the steady state and then its surface temperature was measured using both the IR camera and the liquid crystal. To get the liquid crystal temperature a CCD camera was used and similar lighting conditions to those used in the calibration process were applied.

The heating system consisted of power supply, voltmeter, ammeter, resistor, transformer and several wires that connected with each other like Figure 3 shows. Voltmeter and ammeter were used to get the voltage and electric current transferred to the resistor.

$$Q = U \cdot I [W] \quad (3.1)$$

The area of the surface heated by the resistor was measured with a ruler right after the experiment was done and the power supply was cut off.

$$A_{\text{total}} = H \cdot w [m^2] \quad (3.2)$$

$$A_{\text{total}} = 0,316 \cdot 0,246 = 77,74 \cdot 10^{-3} m^2$$

Parameters H and w represent the height and width of the area heated by resistor.

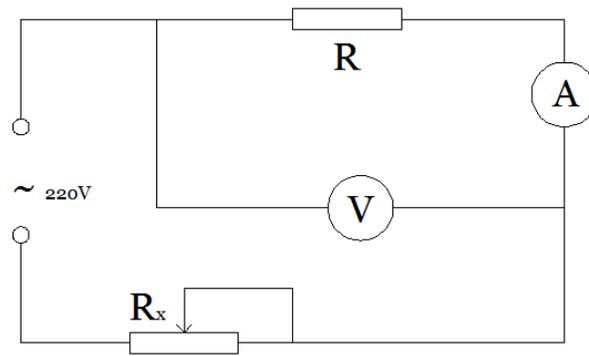


Figure 3: Electric schematic of the experimental work with IRT and LCT

When measuring temperature with the liquid crystal, as said before, the settings were the same as in the calibration process. That is, two fluorescent lamps were mounted vertically right in front of the plexiglass plate to illuminate it. The CCD camera was fixed with a tripod in front of the plexiglass window and perpendicular to the centre of the plate. The CCD camera was then plugged into the computer and specific software helped to process the images and select the specific area. The system of measurement included the CCD camera and the plexiglass plate is covered by a piece of black cloth in order to avoid the influence of other light resources and reflection. Then when the image of liquid crystal was captured, Matlab software was used again to get the temperature distribution of the plate. To do so we applied the functions temperature – hue obtained in the calibration process. One of the three polynomial functions was applied to each pixel of the image taken in order to get its temperature.

For the measurement with the IR camera, a handheld thermal imager was used. As said before, all objects above absolute zero emit infrared radiation, no other lights are allowed except the target surface. Fluorescent lamps were switched off since IR cameras need a dark environment. Also, a black cloth is used to cover the system of measurement to avoid reflection. The reference temperature was measured with a digital thermometer and the emissivity of the surface of the liquid crystal is approximately known to be 0,89. This value is needed to get the actual temperature of the liquid crystal surface.



Figure 4: Picture of LCT

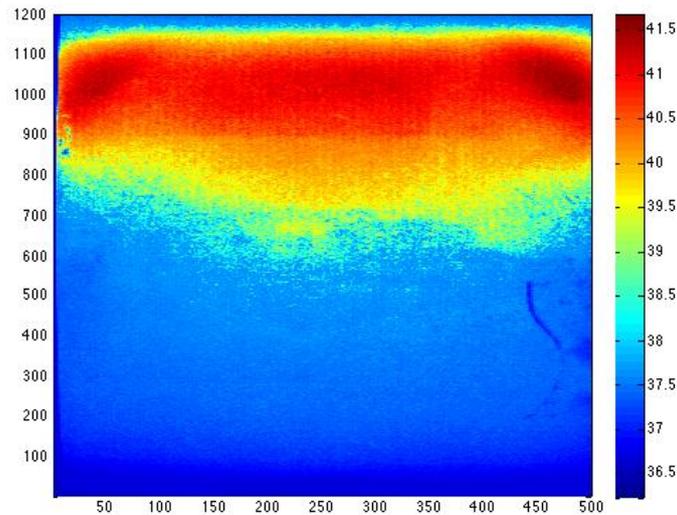


Figure 5: Surface Temperature measured by LCT

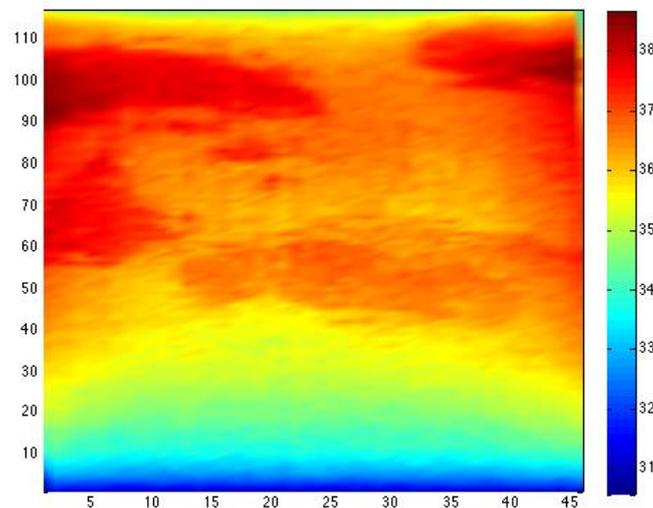


Figure 6: Surface Temperature measured by IRT

Figures 5 and 6 show the temperature distributions of the liquid crystal's surface with both techniques, LCT and IRT respectively. Note that the temperature colour scales are not the same in both images.

A detailed analysis of the results obtained is stated in the next section.

4. Analysis of Results

In the calibration, an adjusted Temperature-Hue curve has been obtained with Matlab according to the data obtained from experiment.

The function (2.2) fits most of the points well. Considering of other unexpected influences like reflection, nicks on the surface of liquid crystal and a changing environment, neglect is allowed especially when hue value is below 20 where liquid

crystal appears to be unsteady. Some critical sections in the polynomial curve obtained are those points where it changes from one expression to the following. It was very hard to attach the functions in a good manner and it resulted impossible to make them fit perfectly.

Since what counts in representing temperature with liquid crystal is hue angle but H, S, I together make up one image; an attempt is done to see if the change of Intensity affects the hue angle of one image. The result shows that 3 times of intensity leads to less than 0.1 °C temperature difference. Details of this attempt are ignored in this report.

In the experiment work, it is actually a natural convection of air on vertical plate. the plexiglass plate was heated until it reached the steady state because the balance of heat produced by resistor and heat transferred to air is needed to calculate the heat using voltage and electric current.

$$Q = U \cdot I$$

$$Q = 51,35V \cdot 0,325A = 16,69W . \quad (4.1)$$

So the surface heat transfer coefficient can be obtained:

$$h = \frac{Q}{A_{\text{total}} \cdot \Delta T} = \frac{Q}{H \cdot w \cdot \Delta T}$$

$$h = \frac{16,69}{0,246 \times 0,316 \times (T_w - T_a)} . \quad (4.2)$$

T_w represents the temperature on the surface of liquid crystal.

T_a represents the ambient temperature which is obtained by digital thermometer as 21 °C

In order to analyse the unexpected influences in this experiment, some figures will be shown. Figures 7 and 9 represent the variation of surface temperature along the central vertical line of the plate according to measures obtained with IRT (figure 7) and LCT (figure 9). Y-axis represents the value of the temperature while X-axis represent the physical height of the points on plate.

Figures 8 and 10 represent the variation of the Heat Transfer Coefficients along the same line. Y-axis represents Heat Transfer Coefficient and, as in figures 7 and 9, X-axis represents the height of the points on plate.

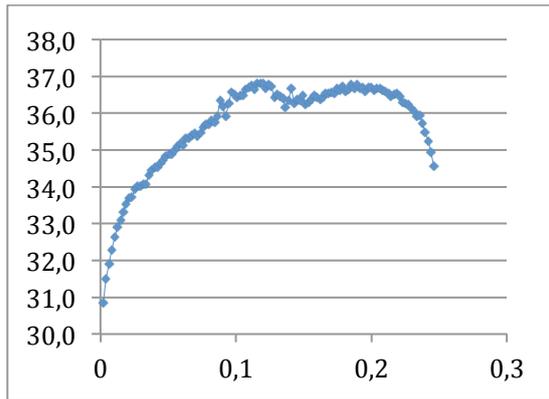


Figure 7: Temperature ($^{\circ}\text{C}$) Changes In Vertical Direction (m) by IRT

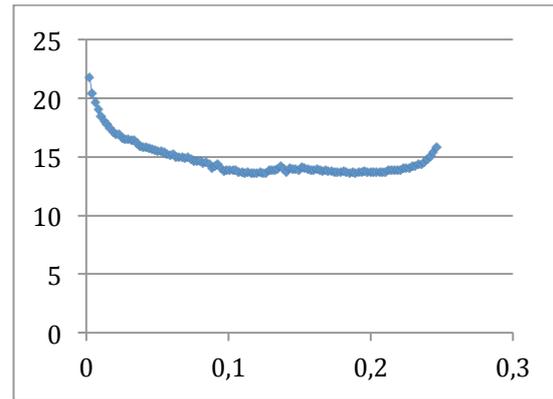


Figure 8: Heat Transfer Coefficient ($\text{W}/(\text{m}^2 \cdot \text{K})$) Changes In Vertical Direction (m) by IRT

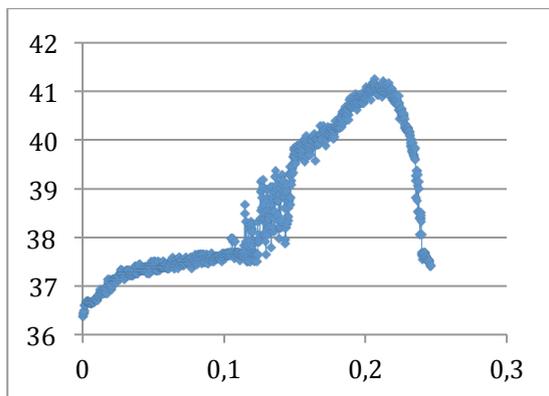


Figure 9: Temperature Changes ($^{\circ}\text{C}$) In Vertical Direction (m) by LCT

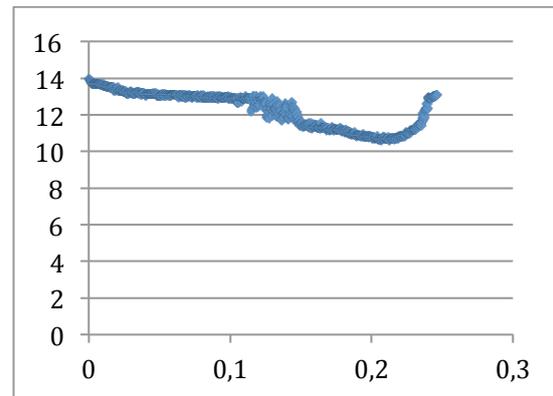


Figure 10: Heat Transfer Coefficient ($\text{W}/(\text{m}^2 \cdot \text{K})$) Changes In Vertical Direction (m) by LCT

As easily found, the intensity of convective heat transfer tend to keep dropping from the bottom to almost top of the plate while temperature of surface in this period keep rising in both IRT and LCT figures.

In fact, as stated before, the experiment carried out with natural convection of air on vertical plate in which thickness of boundary layer keep increasing following the direction where air flows and from bottom to almost top of the plate in this experiment. The boundary layer of air prevents heat transfer so that thicker it gets, less heat can be transferred from the surface to air which leads to higher local temperature appearing in the increasing of temperature in figure 7 and 9. Also, heat transfer coefficient decreases with the deterioration of heat transfer.

It can be noticed that the temperature measured by LCT appears to be more erratic especially during the middle area. The reason of this phenomenon could be that temperature changes a lot with a small increase of hue value during around 37°C to 40°C according to the calibration of liquid crystal, which means the liquid crystal is much more sensitive during this period of temperature.

At the top of the plate, temperature drops and heat transfer coefficient rises because that area is close to the border of heater where heat is produced less. Actually considering of the accuracy of experiments, border areas are ignored in most conditions. Even, heat also transferred through the top edge by convection. In this experiment, heat transferred

to air through all the edges and the backside of the plexiglass plate as well as heat transferred by radiation is ignored. This is to say, heat we calculated is bigger than the heat that actually transfers through the surface.

As seen in figure 7 and figure 9, temperature differs from bottom to top of the plate, which means there is heat transferred in vertical direction. But this part of heat transfer is hard to calculate and its influence in this experiment can be ignored.

Disturbance appeared in these figures could probably be caused by nicks on the surface and air turbulence.

Then experimental Nusselt number can be obtained as well by applying the equation that follows:

$$\text{Nu}_x = \frac{h \cdot x}{K} \quad (4.3)$$

x represents the characteristic length, which changes with height in this experiment. K is the thermal conductivity of air on 21 °C: $K = 0.025 \text{ W}/(\text{m} \cdot \text{K})$.

As laminar natural convection on vertical plate happens in this experiment, theoretical Nusselt number can also be obtained by relation of Grashof number and Prandtl number (G. C. VLIET and C. K. LIU, 1969) which follows:

$$\text{Nu}_x = 0,60(\text{Gr}_x \cdot \text{Pr})^{\frac{1}{5}} \quad (4.4)$$

A modified Grashof number can be obtained by known conditions in this experiment:

$$\text{Gr}^* = \frac{g\beta q_w x^4}{K\nu^2} \quad (4.5)$$

- g represents gravitational acceleration, which is known as $g = 9,8 \text{ m/s}^2$.
- β represents coefficient of volumetric expansion, which is known as $\beta = \frac{1}{T} = \frac{1}{273,15+21 \text{ K}} = 3,4 \times 10^{-3} \text{ K}^{-1}$.
- q_w represents wall heat flux, which can be obtained by $q_w = \frac{Q}{A_{\text{total}}} \text{ (W/m}^2\text{)}$.
- ν represents kinematic viscosity of air on 21 °C, $\nu = 16 \times 10^{-6} \text{ m}^2/\text{s}$.
- Prandtl number of air on 21 °C is known as 0,72.

Following figures show Nusselt number changes in vertical direction and relative error between experimental data and theoretical data.

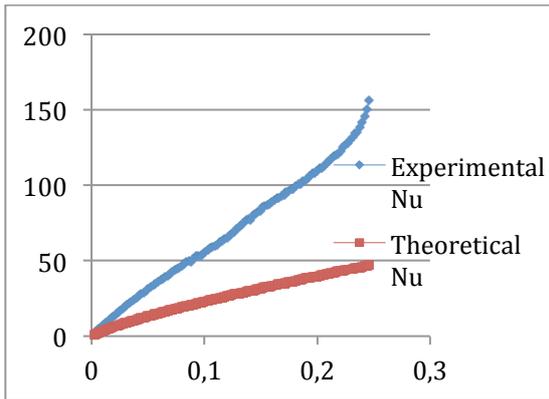


Figure 11: Nusselt Number Changes In Vertical Direction (m) by IRT

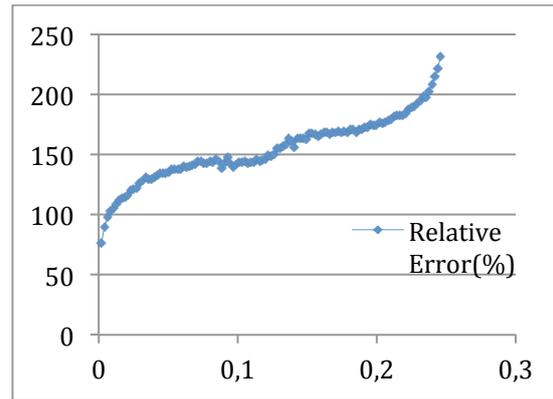


Figure 12: Relative Error (%) Changes In Vertical Direction (m) by IRT

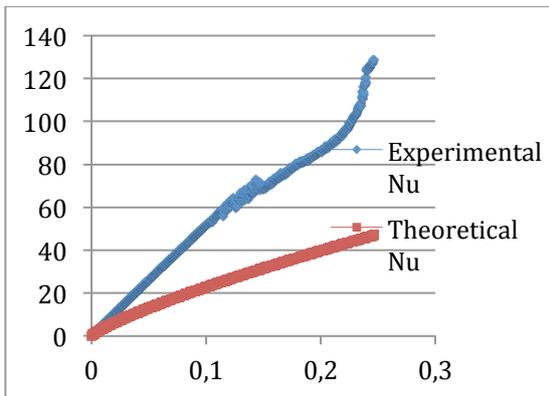


Figure 13: Nusselt Number Changes In Vertical Direction (m) by LCT

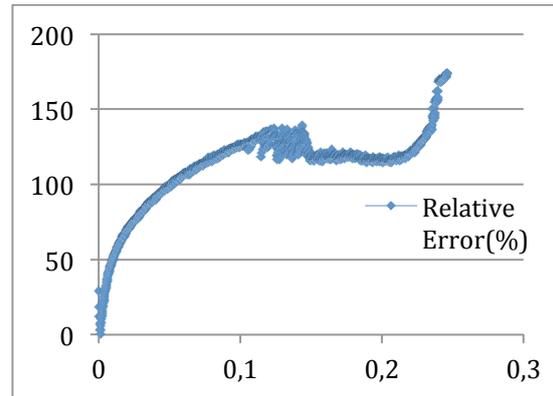


Figure 14: Relative Error (%) Changes In Vertical Direction (m) by LCT

After analysing figures 11 and 13, it can easily be found that experimental Nu is always higher than theoretical Nu at the same height in both IRT and LCT results. As said before, heat calculated is much bigger than the heat that is actually transferred through the surface of the resistor, which leads to higher experimental Nu.

Figures 12 and 14 show the relative error between experimental and theoretical Nusselt number in both IRT and LCT results. Obviously relative error is quite smaller in LCT measurements than in IRT results, which is to say, temperatures measured by LCT are more accurate than those measured by IRT.

According to the literature of IRT, its accuracy is around 2°C in the measurement range between -20°C to 100°C while accuracy of LCT can be around $0,3^{\circ}\text{C}$ under the calibration mentioned in this report. This fact could explain the difference of relative error shown before.

When the IR camera is being used, a specific value for the emissivity of the surface of the liquid crystal is considered but air humidity can also affect the result of IRT. Steam, carbon dioxide and some other kinds of gases can absorb part of infrared radiation, which would result in some percentage of error. If high accuracy is needed this kind of influence can be mostly fixed by measuring the air humidity and considering it when measuring temperatures.

But when using LCT, since the liquid crystal is directly sprayed on the surface, there is no interference between.

IRT as a widely available technique of temperature measurement, have found applications from cryogenic temperature to over 6000K. Since this technique has been mature, the cost of products could be low and products can be made with various features. It does not require any additional source of illumination to operate, which makes them highly attractive in military and surveillance applications.

LCT has been used to investigate various thermal phenomena in a wide variety of applications. These applications include electronics thermal management, gas turbine heat transfer, boiling heat transfer, and fluid temperature measurement. As its advantages over the others as a temperature sensor, LCT can provide detailed temperature distribution (full field mapping) on the test surface, even complex surface. Its resolution can be very high, for instance, $2 \cdot 10^{-4} \text{ }^\circ\text{C}$ temperature change was reported by the noticeable variation of pitch of the helix of the liquid crystal (Zhu and Hieftje, 1989). A resolution of less than 0.1°C is easily obtainable for narrow band liquid crystals through appropriate calibration. Also its short response time can be valued during measurement.

However, liquid crystal can only be used at relatively low temperature, which is lower than 190°C for most liquid crystal. Factors that affect the colour of liquid crystal are various also, such as background lighting, the lighting/viewing characteristics, the camera system, test section properties, etcetera and they need to be kept under control.

5. References

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6. Matlab Scripts

Functions.m

This script calculates the three functions that fit the best all the points (Hue, Temperature) obtained in the calibration process.

%Function 1 Hue from 1 to 92

```
a1 = [6.5646 7.38622492689776 9.2881 10.2932257718102 14.763 53.1712711124943 70.0716  
82.305882656171 85.5662792662622 88.7935606385286];
```

```
b1 = [35.31090228 35.07 35.0511229 35.77 36.0148206 36.52 36.87155032 37.1 37.26 37.53];
```

```
p1 = polyfit(a1,b1,3);
```

```
x1 = 1:1:92;
```

```
y1 = polyval(p1,x1);
```

```
plot(a1,b1,'.');
```

```
hold on;
```

```
plot(x1,y1,'*r');
```

%Function 2 Hue from 92 to 102

```
a2 = [91.9937227378283 92.615 92.8386292323966 93.7621 94.2071437111397 95.078 100.0357  
102.870289736874 104.5611];
```

```
b2 = [38.03 38.43909952 38.51 38.9576742 39.08 39.33181386 40.11509552 40.37 40.50353224];
```

```
p2 = polyfit(a2,b2,3);
```

```
x2 = 92:1:102;
```

```
y2 = polyval(p2,x2);
```

```
plot(a2,b2,'.');
```

```
plot(x2,y2,'*r');
```

%Function 3 Hue from 102 to 135

```
a3 = [95.078 100.0357 102.870289736874 104.5611 108.4534 121.3593 131.672803660695];
```

```
b3 = [39.33181386 40.11509552 40.37 40.50353224 40.81063386 41.532298 41.95];
```

```
p3 = polyfit(a3,b3,3);
```

```
x3 = 102:1:135;
```

```
y3 = polyval(p3,x3);
```

```
plot(a3,b3,'.');
```

```
plot(x3,y3,'*r');
```

Hue_Calibration.m

This script takes a picture and reads its information providing a matrix with RGB data for each pixel. Then transforms it to HIS and it averages the hue value of the whole image. This script was used during the calibration process to get the averaged hue value of each picture that was taken with the CCD camera that was later on associated to a specific temperature.

```
Instr1='image';  
Instr3='.png';
```

```
l=1; %By changing this number the script reads all the images saved  
str2=sprintf('%02d',l);  
Instr=[Instr1 str2 Instr3];  
myImage=imread(Instr);
```

```
[hue s v]=rgb2hsv(myImage); %convert RGB to HSI (range 0~1)  
clear s v; %The saturation s and v values are cancelled in order to shrink the memory.
```

```
hue=255.0*hue;
```

```
[m,n]=size(hue); %m rows and n columns
```

```
%Calculation of the Hue average  
totalhue = 0;
```

```
for i = 1:m;  
    for j = 1:n;  
        totalhue = totalhue + hue(i,j);  
    end  
end  
hueaverage = totalhue/(m*n);
```

LCT_technique.m

This script applies the three functions T1, T2 and T3 or as stated in the Matlab script before p1, p2 and p3 to each pixel in the image taken of the plexiglass plate for the experimental work. It plots the temperature distribution and the Nusselt number distribution of the liquid crystal surface.

```

Instr1='image';
Instr3='.png';

l=1;
str2=sprintf("%02d",l);
Instr=[Instr1 str2 Instr3];
myImage=imread(Instr);

[hue s v]=rgb2hsv(myImage); %convert RGB to HSI (range 0~1)
clear s v; %The saturation s and v values are cancelled in order to shrink the memory.

hue=255.0*hue;

[m,n]=size(hue); %m rows and n columns

%Creates matrix "temp" applying the 3 functions obtained before.
%Transforms Hue value of each pixel into its Temperature
temp = hue;
for i = 1:m;
    for j = 1:n;
        if hue(i,j) <= 91;
            temp(i,j) = polyval(p1,hue(i,j));
        else if hue(i,j) <= 102;
            temp(i,j) = polyval(p2,hue(i,j));
        else
            temp(i,j) = polyval(p3,hue(i,j));
        end
    end
end
end

%Inverts the matrix temp (CCD camera gets it upside-down)
temp2 = temp;
for i = 1:m;
    for j = 1:n;
        temp2(i,j) = temp(m-i+1,j);
    end
end

%Prints image in different colours depending on its temperature
figure('Name','Temperature LCT');
pcolor(temp2);shading interp; colorbar;

%Calculation of the Nusselt number

w = 0.246; %Width of the LCT in meters
l = 0.316; %Length of the LCT in meters
A = w*l; %LCT area
v = 51.3; %Voltage applied to heat the LCT in V

```

```
i = 0.325; %Current flowing through the circuit in A
f = (v*i)/A; %Power used to heat the LCT in W/m2
Tr = 21; %Reference temperature in the lab in Celsius
lamda = 0.02556; %Air thermal conductivity coefficient
```

```
hmatrix = temp2;
Numatrix = hmatrix;
for i = 1:m;
    for j = 1:n;
        dT = temp2(i,j) - Tr;
        hmatrix(i,j) = f/dT;
        Numatrix(i,j) = (hmatrix(i,j)*l)/lamda;
    end
end
figure('Name','Nusselt Number LCT');
pcolor(Numatrix);shading interp; colorbar;
```

IR_technique.m

This script processes the data gathered with the IR camera; it plots both the temperature distribution and the Nusselt number distribution of the image taken with the camera.

```

filename = 'IR camera';
sheet = '2'; %Selecting one of the 2 sheets available in the doc. Sheet 2 includes less pixels than sheet 1
temp = xlsread(filename,sheet);
[m,n] = size(temp);

%Inverts the matrix temp (pcolor function shows it upside-down)
temp2 = temp;
for i = 1:m;
    for j = 1:n;
        temp2(i,j) = temp(m-i+1,j);
    end
end

%Prints image in different colours depending on its temperature
figure('Name','Temperature IR');
pcolor(temp2);shading interp; colorbar;

%Calculation of the Nusselt number

w = 0.246; %Width of the LCT in meters
l = 0.316; %Length of the LCT in meters
A = w*l; %LCT area
v = 51.3; %Voltage applied to heat the LCT in V
i = 0.325; %Current flowing through the circuit in A
f = (v*i)/A; %Power used to heat the LCT in W/m2
Tr = 21; %Reference temperature in the lab in Celsius
lamda = 0.02556; %Air thermal conductivity coefficient

hmatrix = temp2;
Numatrix = hmatrix;
for i = 1:m;
    for j = 1:n;
        dT = temp2(i,j) - Tr;
        hmatrix(i,j) = f/dT;
        Numatrix(i,j) = (hmatrix(i,j)*l)/lamda;
    end
end
figure('Name','Nusselt Number IR');
pcolor(Numatrix);shading interp; colorbar;

```

Lunds Tekniska Högskola, Lund University

Phase Change Materials

Supervisor: Professor Srinivasan Iyengar

Course FKM 065 Project Materials Engineering

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**LUNDS
UNIVERSITET**

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1. Introduction

1.1 Project overview

Have you ever burnt your tongue on a scalding cup of coffee? If you ever did before, this report is the first step on solving that problem. Coffee machines need water at around 100°C to prepare coffee and this means that once you serve it in either a ceramic/paper cup or a thermo mug it is too hot to drink it. The same happens not only with coffee but also with other beverages such as tea that require liquids at a very high temperature to be prepared. The ideal temperature to drink a coffee or a cup of tea is considered to be around 60°C. This means that we have to wait for a while until our beverage decreases its temperature almost 40°C to start drinking it. How would it be if we could manage to cool down these beverages to a specific temperature in a very short period of time and then keep this temperature constant for hours?

1.2 Background

This is just a simple sample from a larger world of thermal energy storage technologies and applications. Roughly, thermal energy storage systems (TES) allow excess thermal energy to be collected, stored and released later on to satisfy the thermal loads of a specific environment. Sustainability has been at the centre of attention for many decades and, considering the increasing price of energy, especially fuel, one of its most challenging areas is related to energy consumption patterns.

Thermal energy storage can be defined in a more technical way by saying that it is the temporary storage of energy at either high or low temperatures. Thermal energy storage systems or methods are divided into three different groups and these are sensible heat storage (SHS), latent heat storage (LHS) and bond energy storage (BES). Sensible heat storage is referred to the system that stores thermal energy without phase change, i.e. increasing or decreasing the temperature of the material being used regardless of its state of matter (solid, liquid or gas) (Ataer 2006). Instead, latent heat storage uses the

phase change between solid and liquid states of matter and its related latent heat to store thermal energy. Finally, bond energy storage, the most recent technology of these three, in some way stores energy in chemical bonds.

Reaching a completely sustainable energy consumption culture has been in the spotlight of many people in the recent years. One of the most representative examples of energy savings achieved using TES systems is related to management of temperature in family dwellings and buildings in general. Many different solutions have been applied in order to keep our homes warm in winter and cold in summer without using energy sources to make stoves, air conditioners and fans work. Some materials used in the past to do so were stones, ceramics and blocks of cast iron (Ataer 2006), (Zondag 2010) and (Pavlov and W. Olessen n.d.).

Thermal energy storage can also be very useful when applied to solar plants. It allows this renewable energy plants to reduce the mismatch between supply and demand of energy at certain times of the day. For instance, when the sun is shining and the solar cells receive the most amounts of heat people may not be using any electric energy to light up their homes. Thereby, TES systems enable these plants to store excesses of produced thermal energy during the daylight to release them during the night. Nevertheless, this project is not aimed at this particular application of TES systems but at a new way of handling hot beverages temperatures.

1.3 Aim of the project

Considering the particular application of the TES system where this report is focused at, the LHS technology is the one that fits best the requirements and needs of the problem that has been raised. This means that a solution that includes the latent heat of a specific material or a compound will be used to solve this issue. Furthermore, among all the available solutions in LHS technology in this project only the thermal storage system that uses phase changing materials (PCM) will be reviewed. These materials appear to be the most interesting solution since they have high storage capacity per volume/mass unit as well as charging and discharging heat from the system where they are applied at

a nearly constant temperature (Abhat 1983). Therefore, the main purpose of this report is to find a solution to the problem raised as well as to briefly evaluate the already existing technologies and products available in the market. Right after this introductory section a chapter about main principles and classification of PCMs will follow. Then in the next chapter named “Selection” some PCM and their properties will be discussed in order to select those that fit best the requirements of the problem stated. Finally in the last chapter an introduction to the main heat transfer methods and principles affecting the particular solutions discussed will be reviewed as well as the encapsulation modes for the PCM that could be applied to these solutions.

1.4 What is in the market now?

Next, nowadays market solutions available will be reviewed. Two are the main products found that are worth to be discussed. These are *Coffee Joulies* and *Temperfect Mug*.

1.4.1 Coffee Joulies

Website: <http://www.joulies.com>

Coffee Joulies are small beans made of stainless steel with PCM inside that melts at 60°C. Each bean is made to work with 11,4 cl of volume. These beans are made to work with any kind of liquid that must stay at a constant temperature of 60°C. The way to use them is by introducing this small beans into the liquid container when it is still hot. In a short period of time the liquid cools down until 60°C and it keeps this temperature for a while.

Price: 59,95\$ (Pack of 5 Coffee joulies) = 43€ = 390 SEK

Strengths and weaknesses (from reviews found of current costumers):

- They are too big so you can fit less liquid in your mug/thermo/cup
- They don't work as they are supposed to do, it requires more joulies than stated on their website to cool down a certain amount of liquid
- They work way better if used with a thermo mug
- You need to wash them after using them, every now and then

- It can be hard to drink from a cup with such big beans sunk inside
- It can be used in any kind of recipients, no need to buy specific cups or mugs



Figure 1. *Samples of Coffee Joulies. Font: www.joulies.com*

1.4.2 Temperperfect mug by Joeveo

Website: <http://joeveo.com>

The solution proposed by Joeveo is a regular 47cl (1 and a half cans of soda) vacuum insulated thermo mug with an extra inner layer of PCM. The PCM melts at 60°C and cools down the liquid in 2 minutes (as stated in their website). This thermo mug is supposed to keep the temperature constant at 60°C for a much longer time than traditional cups and thermos, almost for 3 or 4 hours.

Price: 35\$ = 25€ = 228 SEK

Strengths and weaknesses:

- It has a vacuum layer that keeps it more insulated from the environment. This results in a better behaviour of the PCM layer. It can keep the temperature constant for a longer time.
- It's big enough to fit the coffee or tea needs of most of the potential customers
- It's not completely washable. High temperatures reached in a dishwasher may damage the vacuum and PCM layers.



Figure 2. *Temperfect mug by Joeveo. Font: www.joeveo.com*

1.5 Patents

Regarding the intellectual property rights some patents have been found related to thermal storage of beverages with PCM. These patents are US20130255824 and US7934537 and are attached at the end of the present report. The specific patents for the solutions already existing in the market presented *Coffee Joulies* and *Temperfect mug by Joeveo* could not be found.

1.6 Types of mugs considered

In the present report three different types of mugs are considered and therefore reviewed. These are paper cups, ceramic mugs and metallic thermo mugs. Moreover a fourth type of solution will be analysed: a wrap-up case that could be adapted to any of the three different types of cups/mugs just presented.

2. Phase change materials (PCM)

Among the many different heat storage technologies available today, PCM are classified as Latent Heat Storage (LHS). LHS systems are able to store heat as latent heat of fusion in materials that melt or freeze at a required temperature. In addition to high heat storage performance, PCM provide an isothermal behaviour during the heat exchange, property that makes them suitable for the exposed application.

In their application, PCM are used at solid-liquid phase change. Phase changes involving gas state are not used despite the fact that they usually involve a much higher latent heat. The reason why they are not used is the increase of volume of the gas phase and the difficulty to store.

In order to deploy a PCM system, three components ought to be taken into account:

The heat storage substance itself, a container for the PCM and a heat-exchanging surface. The design of the heat-exchange and the confinement material are significantly important in order to achieve the expected performance of the PCM system.

2.1 Desirable properties for PCM

A broad range of materials that melt at a desired temperature could be identified. Nevertheless, PCM are required desirable thermodynamic, kinetic and chemical properties; as well as economic considerations such as procurement and manipulation costs, and large-scale availability. These desired properties are going to be key in selecting the proper PCM material for the studied application. They are listed and explained below: (Abhat 1983) (Oró, et al. 2012)

- **Thermodynamic properties**
 - **Melting point within the desired temperature range**
 - **High latent heat of fusion** per unit of mass or volume; so that less amount or volume of material is required to absorb and store the required amount of heat.
 - **High specific heat** provides additional heat transfer before the phase change occurs.

- **High thermal conductivity** of both liquid and solid phases, so that temperature gradients required for a certain heat transfer through the PCM are small.
- **Small volume change** during phase transition. The ratio between solid state density and liquid state density should be the closest as possible to one.
- **Congruent melting**; so that the composition of the liquid and the solid phase are the same. Otherwise, the heat transfer capacity would not be constant.
- **Kinetic properties**
 - **High nucleation rate and crystal growth rate** to minimize supercooling during freezing and to assure that the material changes phase at its thermodynamic temperature so that energy is exchanged at the desired temperature. Usually a nucleating agent is needed.
- **Chemical properties**
 - Chemical stability
 - Complete reversible phase change cycle
 - No chemical decomposition
 - No corrosive
 - Non-poisonous, non-flammable and non-explosive
- **Economic properties**
 - Abundant and available
 - Cost-effective
 - Recyclable
 - Easy to conform

2.2 Classification of PCM

According to literature, PCM are classified into families of organic, inorganic materials and their eutectics. Furthermore, organic materials are also divided into paraffin and non-paraffin organics, mainly fatty acids or mixtures. Within inorganic materials,

distinction is done between salt hydrates and metallic materials. This classification is described in figure 3. (Atul Sharma 2007)

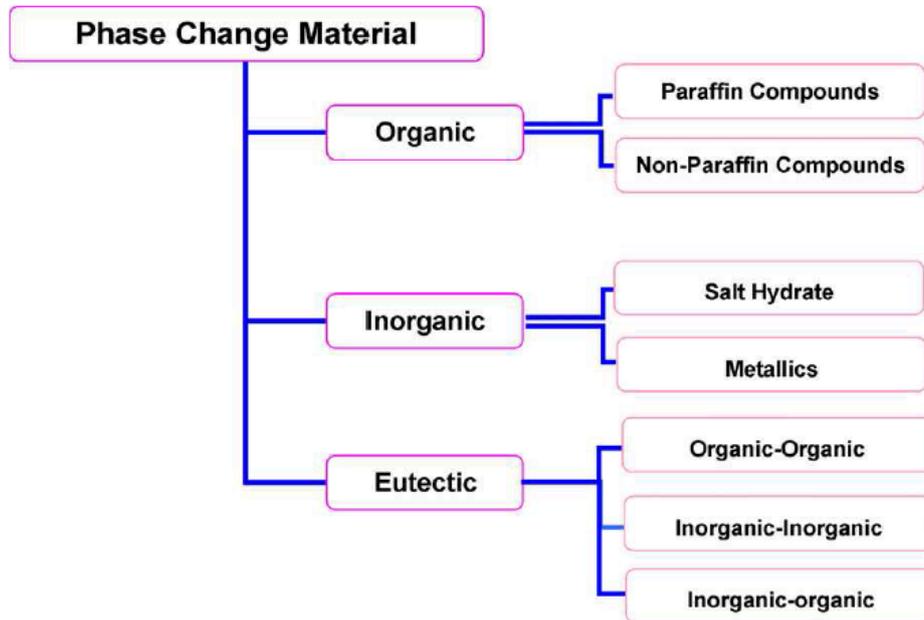


Figure 3. *Classification of PCMs*

2.2.1 Organic phase change materials

In general, organic materials include a wide range of compounds whose molecules contain any carbon. Despite the fact that organic materials have a lower range of latent heat than or instance salt hydrates, organic materials present some characteristics that make them applicable as PCM: they present congruent melting, which means that can be melted and frozen repeatedly without phase segregation, keeping a constant heat of fusion. Organic materials hold self-nucleating properties, which means that they produce very little supercooling when crystallising. Finally they are also non-corrosive and compatible with usual structural materials. A deeper look is required, in order to distinguish between paraffin and non-paraffin organic compounds. (Abhat 1983) (Atul Sharma 2007)

2.2.1.1 Paraffin compounds

Paraffins are known for their waxy consistency at room temperature. They are made up of straight chain of alkanes characterized by C_nH_{n+2} with small amount of branching. The melting point of the paraffins increases with the number of C atoms. For instance, paraffins with 14 to 40 C atoms melt at a temperature between 6 and 80°C. The fact that they are available with a wide range of melting temperatures, together with a reasonably high latent heat, makes them suitable as PCM. Paraffins show reliable and predictable behaviour. They are inert and stable materials below 500°C. Moreover, they show little volume changes when melting. Nevertheless, they show some undesirable properties such as low thermal conductivity, non-compatibility with plastic containers and they are partially flammable. These side effects can be partially overcome with a proper package for the PCM. Table 1 shows some paraffins along with their freezing point and heat of fusion. (Abhat 1983) (Atul Sharma 2007)

Paraffin ^a	Freezing point/ range (°C)	Heat of fusion (kJ/kg)
6106	42–44	189
P116 ^c	45–48	210
5838	48–50	189
6035	58–60	189
6403	62–64	189
6499	66–68	189

Table 1. *Physical properties of some paraffins*

2.2.1.2 *Non-paraffin compounds*

Non-paraffin compounds embrace a lot of materials with very varied properties, such as esters, fatty acids, alcohols and glycols. They are characterised by a high heat of fusion, inflammability, low thermal conductivity, varying level of toxicity and instability at high temperatures. Research is restricted to fatty acids although some other non-paraffin compounds with similar behaviour to fatty acids can also be taken into account.

Fatty acids show high latent heat values, comparable to paraffins. They are characterized by the formula $\text{CH}_3(\text{CH}_2)_{2n}\text{COOH}$ and they also show also reproducible phase changing behaviour and no supercooling when freezing, hence becoming firm PCM candidates. However, their main disadvantage is that they are between 2 and 2.5 times more expensive than paraffins. Table 2 shows the properties of some non-paraffins and table 3 shows some fatty acids together with their melting point and latent heat. (Atul Sharma 2007)

Material	Melting point (°C)	Latent heat (kJ/kg)
Nitro naphthalene	56.7	103
Trimyristin	33-57	201-213
Heptadecanoic acid	60.6	189
α -Chloroacetic acid	61.2	130
Bee wax	61.8	177
Bees wax	61.8	177
Glycolic acid	63.0	109
Glycolic acid	63	109
<i>p</i> -Bromophenol	63.5	86
Azobenzene	67.1	121
Acrylic acid	68.0	115
Dinito toluent (2,4)	70.0	111
Phenylacetic acid	76.7	102

Table 2. *Physical properties of some non-paraffins*

Material	Formula	Melting point (°C)	Latent heat (kJ/kg)
Acetic acid	CH_3COOH	16.7	184
Polyethylene glycol 600	$\text{H}(\text{OC}_2\text{H}_4)_n\text{OH}$	20-25	146
Capric acid	$\text{CH}_3(\text{CH}_2)_8\text{COOH}$	36	152
Eladic acid	$\text{C}_8\text{H}_7\text{C}_9\text{H}_{16}\text{COOH}$	47	218
Lauric acid	$\text{CH}_3(\text{CH}_2)_{10}\text{COOH}$	49	178
Pentadecanoic acid	$\text{CH}_3(\text{CH}_2)_{13}\text{COOH}$	52.5	178
Tristearin	$(\text{C}_{17}\text{H}_{35}\text{COO})\text{C}_3\text{H}_5$	56	191
Myristic acid	$\text{CH}_3(\text{CH}_2)_{12}\text{COOH}$	58	199
Palmitic acid	$\text{CH}_3(\text{CH}_2)_{14}\text{COOH}$	55	163
Stearic acid	$\text{CH}_3(\text{CH}_2)_{16}\text{COOH}$	69.4	199
Acetamide	CH_3CONH_2	81	241
Methyl fumarate	$(\text{CHCO}_2\text{NH}_3)_2$	102	242

Table 3. *Physical properties of some fatty acids*

2.3.2 Inorganic phase-change materials

Inorganic phase-change materials include salt hydrates and metallic materials. This kind of materials shows no supercooling and their latent fusion keeps constant after multiples cycles.

2.3.2.1 Salt hydrates

Salt hydrates are characterized by the formula $AB \cdot nH_2O$, where AB is an inorganic compound that has been hydrated with n molecules of water per unit of salt. Salt hydrates show a high volumetric latent heat. Moreover, salt hydrates present a relatively high thermal conductivity and small volume changes when melting. They are not very corrosive, compatible with plastics and not very toxic.

Nevertheless, the major problem with salt hydrates is that they usually melt incongruently. Salt hydrates melt to a lower hydrate of the salt or to its anhydrous form. As the water of crystallisation is not enough to dissolve all the solid phase, the result of the melting is a supersaturated solution, where the solid salt settles down of the container due to its higher density. This phenomenon is irreversible and it is called decomposition: during the freezing, the solid phase does not combine with the saturated solution to form the original salt hydrate. In addition, hydrated salts suffer supercooling when freezing, due to poor nucleating properties. Incongruent melting can be solved in different ways such as by mechanical stirring, by encapsulating PCM to reduce separation, by adding thickening agents to improve the suspension of salts and hinder their precipitation, by using excess water to prevent supersaturation or by modifying the composition of the hydrated salt to make it congruent. On the other hand, some methods are proposed in literature in order to prevent supercooling and enhance nucleating properties, such as the addition of nucleating agents, using a “cold finger” or rough metallic heat exchanging surfaces to promote heterogeneous nucleation. (Abhat 1983) (Atul Sharma 2007)

Table 5
Melting point and latent heat of fusion: salt hydrates

Material	Melting point (°C)	Latent heat (kJ/kg)
FeCl ₃ ·2H ₂ O	56	90
Ni(NO ₃) ₂ ·6H ₂ O	57.0	169
MnCl ₂ ·4H ₂ O	58.0	151
MgCl ₂ ·4H ₂ O	58.0	178
CH ₃ COONa·3H ₂ O	58.0	265
Fe(NO ₃) ₂ ·6H ₂ O	60.5	126
NaAl(SO ₄) ₂ ·10H ₂ O	61.0	181
NaOH·H ₂ O	64.3	273
Na ₃ PO ₄ ·12H ₂ O	65.0	190
LiCH ₃ COO·2H ₂ O	70	150
Al(NO ₃) ₂ ·9H ₂ O	72	155
Ba(OH) ₂ ·8H ₂ O	78	265
Mg(NO ₃) ₂ ·6H ₂ O	89.9	167
KAl(SO ₄) ₂ ·12H ₂ O	91	184
MgCl ₂ ·6H ₂ O	117	167

Table 4. *Physical properties of some salt hydrates*

2.3.2.2 Metallic compounds

This group includes metallic compounds with low melting points. Their major problem is their high density, which makes them heavy materials. However, they show high volumetric latent heat, although they show low latent heat per unit mass; high-thermal conductivity and low specific heat. Table 5 shows some metallic compounds considered as PCM, along with their melting points and latent heat per unit mass. [c]

Material	Melting point (°C)	Latent heat (kJ/kg)
Gallium–gallium antimony eutectic	29.8	–
Gallium	30.0	80.3
Cerrolow eutectic	58	90.9
Bi–Cd–In eutectic	61	25
Cerrobend eutectic	70	32.6
Bi–Pb–In eutectic	70	29
Bi–In eutectic	72	25
Bi–Pb–tin eutectic	96	–
Bi–Pb eutectic	125	–

Table 5. *Physical properties of some metallic compounds*

2.3.3 Eutectics

Eutectic compounds are a mixture of two or more components that present a fixed melting point, which is the lowest point where a mixture of its components could ever melt. Thus, they freeze and melt congruently forming a mixture of the component crystals. Eutectics melt without segregation too. Table 7 shows some organic and inorganic compounds used as PCM, together with their composition, melting point and latent heat per unit mass. [c]

Material	Composition (wt.%)	Melting point (°C)	Latent heat (kJ/kg)
CaCl ₂ ·6H ₂ O + CaBr ₂ ·6H ₂ O	45 + 55	14.7	140
Triethylolthane + water + urea	38.5 + 31.5 + 30	13.4	160
C ₁₄ H ₂₈ O ₂ + C ₁₀ H ₂₀ O ₂	34 + 66	24	147.7
CaCl ₂ + MgCl ₂ ·6H ₂ O	50 + 50	25	95
CH ₃ CONH ₂ + NH ₂ CONH ₂	50 + 50	27	163
Triethylolthane + urea	62.5 + 37.5	29.8	218
Ca(NO ₃) ₂ ·4H ₂ O + Mg(NO ₃) ₂ ·6H ₂ O	47 + 53	30	136
CH ₃ COONa·3H ₂ O + NH ₂ CONH ₂	40 + 60	30	200.5
NH ₂ CONH ₂ + NH ₄ NO ₃	53 + 47	46	95
Mg(NO ₃) ₂ ·6H ₂ O + NH ₄ NO ₃	61.5 + 38.5	52	125.5
Mg(NO ₃) ₂ ·6H ₂ O + MgCl ₂ ·6H ₂ O	58.7 + 41.3	59	132.2
Mg(NO ₃) ₂ ·6H ₂ O + MgCl ₂ ·6H ₂ O	50 + 50	59.1	144
Mg(NO ₃) ₂ ·6H ₂ O + Al(NO ₃) ₃ ·9H ₂ O	53 + 47	61	148
CH ₃ CONH ₂ + C ₁₇ H ₃₅ COOH	50 + 50	65	218
Mg(NO ₃) ₂ ·6H ₂ O + MgBr ₂ ·6H ₂ O	59 + 41	66	168
Napthalene + benzoic acid	67.1 + 32.9	67	123.4
NH ₂ CONH ₂ + NH ₄ Br	66.6 + 33.4	76	151
LiNO ₃ + NH ₄ NO ₃ + NaNO ₃	25 + 65 + 10	80.5	113
LiNO ₃ + NH ₄ NO ₃ + KNO ₃	26.4 + 58.7 + 14.9	81.5	116
LiNO ₃ + NH ₄ NO ₃ + NH ₄ Cl	27 + 68 + 5	81.6	108

Table 7. Physical properties of some eutectic compounds

3. Selection

Among all the Phase Changing Materials mentioned in the previous tables, some of the materials that are applicable for the studied application have been selected and listed in table 8, together with some of their qualitative properties. The table has been fulfilled after extensive research and, due to the specificity of some of the materials, there is several data missing. Thus, material selection process is going to be based on the data available.

Type	Sub Type	PCM	Melting Point [°C]	Latent Heat [KJ/kg]	Latent heat [Kj/l]	Specific heat [J/kgK]	Thermal conductivity (W/m *K) (solid)	Solid density [g/cm3]	Liquid density [g/cm3]
Organic	Paraffin	6035*	58-60	189	170,1	2100	0,21	0,9	0,76
		6403*	62-64	189	170,1	2100	0,21	0,9	0,79
	Non Paraffin	Heptaudecanoic acid / Margaric acid	60,6	189	161,2	--	--	0,853	
		alpha-Chloroacetic acid	61,2	130	205,4	1916	--	1,58	1,3703
		Bee wax	61,8	177	169,6	3400	0,25	0,958	0,822
		Palmatic acid (fatty acid)	55	163	139	1844	0,144	0,853	0,847
		Myristic acid (fatty acid)	58	199	171,6	1600	--	0,862	0,844
Inorganic	Salt hydrates	Ni(NO3)2·6H2O	57	169	346,5	--	--	2,05	--
		MnCl2·4H2O	58	151	303,5	--	--	2,01	--
		Fe(NO3)2·6H2O	60,5	126	189	--	--	1,5	--
		CH3COONa·3H2O	58	265	404,9	--	--	1,528	--
Metal		Cerrolow eutectic	58	90,9	799,9	--	--	8,8	--
		Bi-Cd-In eutectic	61	25	225,3	--	--	9,01	--
Eutectic	Inorganic - Inorganic	Mg(NO3)3·6H2O + MgCl2·6H2O (50-50)	59,1	144		--	--	--	--
		Mg(NO3)3·6H2O + MgCl2·6H2O (58,7-41,3)	59	132,2	215,5	--	0,678	1,63	1,55
	Inorganic - Inorganic	Mg(NO3)3·6H2O + Al(NO3)2·9H2O (53-47)	61	148	249	--	--	--	--

Table 8. Selected Phase Changing Materials with melting point around 60°C
 * Manufacturer of technical grade paraffins: Ter Hell Paraffins, Hamburg, FRG.

Referring to the selection criteria stated in previous sections, the first thing to bear in mind when choosing a phase changing material is the range of temperature in which it can be used. In the case of study, the required performance temperature is around 60°C, as it is the optimal temperature in which coffee is most enjoyed. Hence, in table 8 only materials with fusion temperature around 60°C have been selected. The second requirement has to do with the amount of heat that needs to be exchanged between the coffee and the phase change material. In order to get an idea of the amount of heat exchange taking place, a transient thermal study is necessary, which involves tough calculations or simulations. Instead of that, a quantitative analysis is going to be carried out. Heat latent specific heat of fusion gives an idea of the amount of material that is going to be required to fulfil specifications. It can be understood in terms of volume or in terms of mass, related by the density of the material. For instance, as it can be seen in chart 1 and due to their high density, metallic metals have a very high specific latent heat of fusion per unit volume, but low per unit mass, which makes them not a proper selection for the studied application because it would become too heavy. Chart 1 shows the correlation between specific heat of fusion per unit mass and per unit volume for the chosen materials.

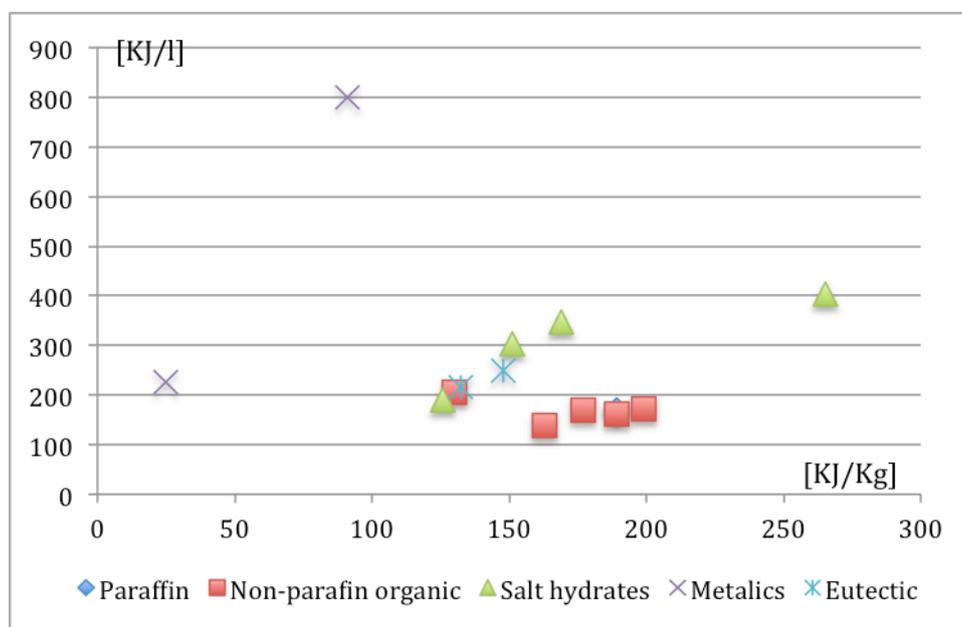


Chart 1. PCM and their specific latent heat per unit mass and unit volume

It is possible to see how mainly, materials from the same families are found close within the chart. According to this chart, $\text{CH}_3\text{COONa}\cdot 3\text{H}_2\text{O}$ is the material that most properly maximizes both mass and volume specific heat of fusion. However, little information has been found on this salt hydrate, despite its density. As a salt hydrate, it is very likely that its nucleation properties are very poor and that it suffers supercooling. Thus a nucleating agent should be utilised. Moreover salt hydrates are the only materials in this table that present incongruent melting. On the other hand, it is neither corrosive nor toxic, chemically stable and it is compatible with plastic containers. Salt hydrates in general have good volume specific performance, due to their higher density compared to organic materials. Paraffins and non-paraffins have similar behaviours as organic materials, with fairly high mass specific latent heat, slightly higher in average than all selected salt hydrates except $\text{CH}_3\text{COONa}\cdot 3\text{H}_2\text{O}$. Another important parameter is the volume variation between solid and liquid state. This can be calculated from the variation between solid density and liquid density. However, in the major volume change among these materials is around 15%, which does not become a problem in the scale of application and making the container bigger can easily solve it. Among all these materials, there is one material which is very commonly commercialised and from which it has been easy to find all kind of data. It is bee wax and as a non-paraffin organic material, with a fairly high specific latent heat per unit of mass. As an organic material, it presents good nucleating properties, congruent melting and chemical stability. The only drawback is that it is a flammable material, with its flash point at 204°C , which should not be dangerous. In addition, bee wax should only be melted in stainless steel, plastic, or tin-plated containers. In addition, it has the highest specific heat, which reduces the necessary amount of material required as well. It is commercialised for a price around 0,25€/g.

Some research in already commercialised solutions has been done. There are some companies nowadays that commercialise phase changing materials for any type of application within a wide range of temperatures. These materials are listed in table 9, together with their properties and the company that commercializes them. Unluckily, no further information such as composition of these products and price has been accessible, as they provide a budget based on volume demand. However, according to the different prospects, they all coincide in being renewable, biodegradable, non-toxic and

chemically stable products, with good and stable performance after a great number of cycles.

Company	Product	Melting Point [°C]	Latent Heat [KJ/kg]	Latent heat [Kj/l]	Specific heat liq. [J/kgK]	Solid density [g/cm3]//[Kg/l]
PureTemp	PureTemp 60	61	230	200,1	2040	0,87
	PureTemp 63	63	199	167,16	1990	
Phase Changing Products PTY LTD	PC58	58	226	327,7	4580	1,45
Phase Changing Material Products Limited	S58 (salt hydrate)	58	145	218	2550	1,505
	A60 (organic)	60	145	132	2220	0,91
	A60H (organic)	60	212	170	2150	0,8
	A58 (organic)	58	132	120	2220	0,91

Table 9. *Currently commercialized Phase Changing Materials with melting point around 60°C.* (PureTemp 2014) (Phase Changing Products PTY LTD 2014) (Phase Changing Material Products Limited 2014)

These products have been plotted in chart 2, together with the previously analysed products, and they seem to have similar characteristics to paraffins and salt hydrates. There is one product that overcomes the other materials. PC58, from Phase Changing Products PTY LTD, shows a high specific latent heat per unit of mass and volume. Moreover, it shows the highest specific heat among all the studied materials, which makes it the most optimal material for a PCM application in terms of quantity of material required. However, price is not taken into account.

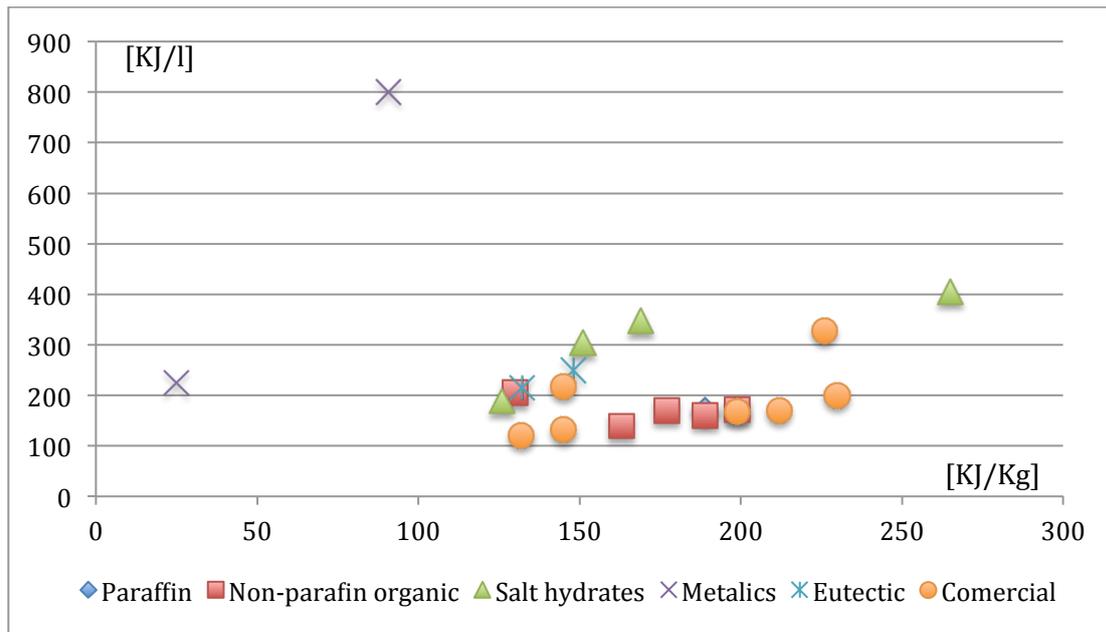


Chart 2. PCM together with commercial PCM and their specific latent heat per unit mass and unit volume.

Thus, in this section, the suitability of different phase changing materials has been discussed and analysed. Lack of information makes it difficult to choose one particular material, but some materials that would properly meet the requirements, are currently available.

4. Dimensioning

4.1 Heat transfer

Heat transfer analysis becomes especially important in this project since the amount of heat exchanged between coffee, the PCM and the environment will determine the temperature behaviour of the beverage. To begin with, a brief introduction about different heat transfer means and mechanisms will be expounded. Then the heat transfer conditions and mechanisms of the present project will be set. However, due to the complexity of the topic and the difficulties encountered when trying to create analysis with several software applications available no simulations will be done and this section will end there.

Heat transfer can be defined as the energy exchange between bodies, whose state of matter can be solid, liquid or gas, due to an existing temperature gradient. This transfer

can occur in three different modes, being all of them simultaneously or not. These three modes or mechanisms are: conduction, convection and radiation. Next, their fundamentals are briefly exposed.

4.1.1 Conduction

Thermal conduction can take place through both solids and fluids but usually thermal conduction through fluids may be considered negligible. In solids, heat energy is transferred by the movement of electrons within the lattice of fixed molecules (Olukayode 2011). Particles forming a hot body vibrate due to its temperature and collide with the cooler particles nearby that thereafter start to vibrate and therefore increase their temperature. The empirical equation that follows was proposed by Fourier and determines the rate at which heat energy is transferred by conduction:

$$\text{Vectorial: } \frac{\vec{q}}{A} = -\bar{\lambda} \cdot \bar{\nabla}T(\vec{r}, t)$$

$$\text{Monodimensional: } \left[\frac{q}{A} \right]_x = -\lambda \cdot \frac{dT}{dx}$$

Where $\left[\frac{q}{A} \right]_x$ is the rate of heat flow per area in x direction expressed in $\left[\frac{W}{m^2} \right]$, λ is the thermal conductivity coefficient of the material considered expressed in $\left[\frac{W}{mK} \right]$ and $\frac{dT}{dx}$ is the thermal gradient in the x direction expressed in $\left[\frac{K}{m} \right]$.

Thermal conductivity coefficient λ measures how fast heat energy can be transferred through the material considered. The higher its value is, the faster heat is transferred from hot to cold areas.

4.1.2 Convection

Convection is the process by which internal energy or heat is transferred thanks to the movement of a fluid. This mode of heat transfer can only take place within fluids that allow their particles to move without breaking the physical continuity of the material. When a fluid is heated by an object, its closest layer becomes warmer and since hot

fluids are less dense than cold fluids this results in a convective flow of fluid from the hot area to the cold area in a movement known as buoyancy effect. Convection process is highly dependent on the movement of the fluid and thereby it can be classified into two different modes: natural or free convection and forced convection. This classification stands for the way in which the fluid motion is generated.

Natural convection occurs only when the fluid is moved by natural means, i.e. by the temperature gradient between two different zones. Instead, when the movement is created by other means such as fans or pumps the heat transfer is known as forced convection (Olukayode 2011) and (Martinez 1992).

The main law that describes this mechanism is Newton's cooling law and is expressed as follows:

$$q = h \cdot A \cdot (T_0 - T_\infty)$$

Where q is the rate at which heat is transferred [W], h is the convective heat transfer coefficient, A the cross sectional area whereby heat transfer occurs (perpendicular to the heat flux) and $(T_0 - T_\infty)$ the temperature gradient between the bodies and/or fluids considered.

4.1.3 Radiation

Finally, radiation is a heat transfer mode based on the emission of electromagnetic waves. All bodies above whose temperature is above absolute zero emit electromagnetic waves that transfer energy. The particularity of this mechanism is that it does not require any material medium to transfer energy, contrary to conduction and convection that need a solid or fluid medium to make heat transfer happen. The amount of energy exchanged between two bodies depends on the distance between them, their temperature, their relative orientation, their area and the wavelength of the radiation emitted.

The rate of radiation emitted by a black body due to its temperature is given by Stefan-Boltzmann's law:

$$E = \sigma \cdot T^4$$

Where σ is Stefan-Boltzmann constant and equals $5,67 \cdot 10^{-8} \frac{W}{m^2K^4}$.

The amount of energy exchanged between two bodies depend on many different things that will not be further analysed since heat transfer by radiation can be underestimated in the particular scope of the present project.

4.1.4 Heat transfer conditions

In the present study some specific heat transfer conditions must be set so that an accurate but efficient and easily comprehensive analysis of the thermal behaviour can be performed. The system considered in this project to be analysed is described next. This system is formed by several elements:

- Mug (ceramic or paper) / Thermo mug (metallic)
- PCM (as a layer or microencapsulated in very small spheres)
- Liquid poured inside the mug (usually coffee)
- Environment (air at room temperature and surface where mug leans)

The main purpose of this chapter is to set the thermal conditions that apply to the heat transfer process of a traditional mug, regardless of its base material (ceramics or paper), as well as a metallic thermo mug. The thermal process that has to be analysed and studied is the transient state that starts when coffee is poured inside a mug and ends when it gets too cold to be drinkable.

Apart from their main material these products would have a PCM as a manufacturing material either in a thin layer located within the main material or in microcapsules. However, the encapsulation of the PCM, i.e. the way it is contained inside the structure of the cup preventing it from spreading around the mug and getting mixed with the coffee, will be discussed in the next chapter. The conditions related to heat transfer are different in these two cases. If the PCM is used as a layer between two walls of the main material the thermal analysis must consider conduction between these two different materials. Instead, if the PCM is encapsulated in micro spheres that are then mixed with the main structural material such as ceramics to create a uniform material then the thermal analysis is easier.

Main heat transfer modes considered are as follows:

- Radiation is omitted since temperatures reached by the different elements of the system are reasonably low
- Convection is considered between coffee and the air of the environment as well as with mug's walls and base
- Convection is also considered between mug's external faces and environment's air
- Conduction through different materials is considered when mug's walls are made of several layers
- Conduction is also considered between mug's base and the support surface where the mug leans

In every convection process considered natural convection is applied since movement of air surrounding the mug is created only by natural means.

Mug's handle must be considered as a fin and should be analysed as such.

The thermal analysis of the process is done as a transient study where three main stages occur:

- 1) Mug is at room temperature and coffee is poured inside it at 100°C. Coffee decreases its temperature by transferring heat to both environment's air (convection) and mug (convection). Mug's walls, including PCM, increase their temperature from room temperature up to PCM's melting point (usually 60°C).
- 2) PCM starts melting at a constant temperature absorbing coffee's heat and thereby decreasing its temperature. Coffee reaches the ideal temperature of 60°C.
- 3) PCM, while solidifying, releases heat to coffee as it gets colder due to heat transfer with the environment keeping its temperature almost constant at 60°C. Eventually, when PCM is fully solidified it starts getting colder together with coffee.

In order to be able to analyse this thermal processes described some coefficients and several variables are required to be known. These are:

- Thermal conductivity coefficient of PCM and mug's main material (ceramic or paper) or metal in case of thermo mugs

- Convective heat transfer coefficient of coffee and environment's air
- Mug's or thermo mug's dimensions such as diameter, height and wall thickness
- Handler's dimensions
- Thickness of the PCM layer (if not microencapsulated)
- PCM total mass
- Coffee total mass
- Environment's air temperature and support surface's temperature
- PCM's heat capacity in solid state and latent heat of fusion

As said before, no further analysis of the heat transfer process will be done in this report since its main spotlight are the phase change materials (PCM), their fundamentals, characteristics, applications and selection for this particular case.

4.2 Encapsulation

Once the several phase change materials available nowadays within the materials engineering industry have been presented an analysis should be made, regarding their implementation into each of the product types described in the introductory chapter of the present report.

The main issue to be discussed is how the PCM should be applied to those types of mugs so that the thermal energy storage and the subsequent heat transfer is properly enhanced and as much efficient as possible. It should also be beard in mind that the final solution must be easily manufacturable as well as comfortable for the final costumer.

Several methods to encapsulate PCM have been studied and applied in the last three or four decades. *Encapsulation* or more broadly *storage* of PCM is the way they are physically isolated from the surrounding materials. Roughly, PCM can be stored/encapsulated in several ways such as plastic films, microcapsules, macrocapsules, metal pipes and many more.

In the present report only microencapsulation in micro spheres and plastic coatings/films will be considered since these solutions are those that fit best the different types of mugs presented.

4.2.1 PCM inner layer

The first and most intuitive way of encapsulating the PCM in the systems considered is by creating a thin layer between two walls made of the main construction material (ceramics or metal). That is a *sandwich* where external layers would be made of either ceramics (when ceramic mugs are considered) or metal (for the thermo mug) whereas the inner layer would be the PCM. This solution has already been successfully applied in the *Temperfect Mug* presented in the introductory chapter of the present report and seems to be the easiest way of applying a PCM to a metal thermo mug. However, it has not been applied yet to any kind of ceramic mugs or containers and the possibility of building this type of mugs with a couple of ceramic layers should be studied in depth together with porcelain manufacturers.

4.2.2 Microencapsulation

Microencapsulation refers to a process where droplets of liquids, solids or gases are coated by thin films known as coatings that protect the core material (Sheu and Rosenberg 1995). Therefore microencapsulated PCM are small portions of material coated by thin films forming very small capsules with a diameter of around 10 to 1000 microns.

Nowadays a very varied range of coating materials exists and all of them should meet the characteristics that follow (Khin 2003):

- Stable to environmental conditions
- High strength, certain flexibility and thermal stability within its application's temperature range
- Not corrosive to container materials
- Non reactive with the PCM core material

Considering the application of the PCM in this project some other characteristics should be met such as biodegradability, low cost and compatibility with both core and surrounding materials.



Figure 4. *Samples of different size phase change materials at Entropy Solutions.* Font: Pioneer Press (Scott Takushi)

The main advantage of this encapsulation method is that once the PCM is coated by a film the whole sphere does not increase its volume when heated. Thereby, the size of the capsule must take into account that as the PCM is heated up it eventually melts and thus its volume increases. This leads to conclude that coatings must be slightly bigger than the amount of solid PCM used as a core material.

In the present report microencapsulated PCM is only considered to be applicable to ceramic mugs. In order to do so they must be mixed with ceramic dust before mugs are manufactured. Nevertheless, ceramic mugs are usually produced by casting processes that may require rather high temperatures. These high temperatures could lead the coating materials up to their melting point as well as to boil the PCM core material. It should therefore be considered to use other materials instead of ceramics such as plaster and gypsum to be mixed with PCM microcapsules. Both plaster and gypsum enhanced with PCM microcapsules have been previously tested as building materials in order to increase the thermal comfort and thus energy savings in buildings (Zamalloa, et al. n.d.) and (Shukla, Fallahi and Kosny 2012). Manufacturing processes of plaster and gypsum and its by-products do not require high temperatures, so that PCM microcapsules could be mixed with it to create a matrix to be later used as a main building material of mugs.

It must be studied together with manufacturing companies whether it is possible to build mugs with such materials or not.

4.2.3 Wrap-up case

The last encapsulation method to be proposed and that suits best paper type mugs is the wrap-up case. It consists of a modular and flexible plastic sheet filled with PCM. This modular case is based on the idea behind metallic watchstraps, which can be easily adjusted to someone's wrist by adding or removing some *modules*. The wrap-up case would be formed by several modules attached to each other with magnets or mechanic joints so it could be adapted to fit any kind of mug, regardless of its size and shape. Each module would have some amount of PCM in it. The wrap-up case could be used inside or outside the mug, i.e. in direct contact with the liquid, where heat transfer would be more efficient, or as an external case.

The wrap-up case concept has been used in many different applications before. One of the most representative among all of them and highly related to the topic of the present report is champagne cooler bag. These bags contain some gel inside them that right after being cooled in the freezer keep their temperature low while extracting heat from the champagne bottle they wrap up.



Figure 5. *Wrap-up cooler bag for champagne bottles*

5. Conclusions

As a latent heat storage system that taps into the concept of latent heat of fusion to store energy PCMs have an isothermal behaviour during the heat exchange, property that makes them suitable for the exposed application.

Besides the melting point within the desired temperature range, in order to find an appropriated PCM material for a particular application, some thermodynamic, kinetic and chemical properties have to be fulfilled, as well as economic considerations such as procurement and manipulation costs and large-scale availability.

PCM are classified into families of organic materials, including paraffins and non-paraffin compounds such as fatty acids; inorganic materials, including salt hydrates and metallics; and the eutectics of organic and inorganic materials.

After analysing the properties of the different groups of materials and presenting some already commercialized alternatives, a wide range of alternatives can be considered for this application. However, we arrived to the conclusion that we lack of information coming from experimental heat transfer analysis or simulations to dimension the problem and tighten the range of optimal materials. Economic data would also play a role in the final election.

Regarding the heat transfer conditions discussed every product solution should consider mainly convection and conduction. The PCM encapsulation method chosen must ensure a proper heat transfer between the hot beverage and the PCM so that heat can be extracted and then released as fast as possible. In order to carry out a simulation analysis many data must be known such as thermal conductivity coefficient of every material involved in the heat transfer process as well as convective heat transfer coefficients of air and the hot beverage.

The amount of PCM used in the mug will depend on how fast and how much liquid needs to be cooled down to 60°C and how long it is necessary to keep it at that temperature. Moreover, the encapsulation method chosen will affect the heat transfer conditions and its effects, decreasing PCM performance, should be considered.

The encapsulation or storage of the PCM will depend on how mugs can be manufactured and which materials are used in that process. As discussed in chapter 4 three are the main solutions available that could fit the types of mugs considered: microencapsulation in micro spheres, inner layer of PCM

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