

# Polymorphism of chocolate

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The purpose of this project was to study the different polymorphic forms of chocolate using calorimetric methods. The main component of chocolate is cocoa butter. Cocoa butter is composed of triglycerides which are in motion in the liquid state. When these triglycerides solidify, they will form structures. If these structures are regular, we can speak of crystallization. The characteristic of cocoa butter is that it can crystallize in different structures: this is called polymorphism. There are six polymorphic forms of cocoa butter. The desired form, called form V, has a melting point between 32 and 34°C. This form has all the benefits of a good chocolate: the chocolate is smooth, shiny, easy to break, crunchy. Chocolate with the form V melts only in the mouth and has a pleasant taste and texture. Form V is achieved by tempering chocolate. This is a key step in the preparation of chocolate. Tempering allows to obtain the most stable crystalline form by following specific temperature sequences. To achieve the goal of this project, several products were analyzed; cocoa butter, couverture chocolate (which must contain at least 32% cocoa butter) and homemade chocolate to try to observe other polymorphic forms.

A DSC (Differential Scanning Calorimetry) was used to determine the cocoa butter melting point. The DSC measures the heat transfer between a system and the environment during a process like fusion. During this project, a DSC with a heat flow mode was used. The apparatus has one oven which contains the reference and the sample. The difference of the heat flow between oven / sample and oven / reference is measured.

An Easy-Max, developed by Mettler Toledo, was also used to determine the cocoa butter melting point. The Easy-Max is a calorimetric reactor and it is very commonly used for a scale-up development. It is possible to work in three modes: isoperibolic, quasi-adiabatic or with temperature ramp. In this project, the Easy-Max presents an advantage because it is possible to work in a wide temperature range (-40 to 180°C).

The results obtained with the DSC are inconclusive. Cocoa butter and homemade chocolate were analyzed. Our DSC does not have a cryostat to cool the device. Therefore, the temperature of the DSC does not drop below 25°C and the melting temperatures found are not correct. Cocoa butter and homemade chocolate are already soft when introduced into the DSC because it has a relatively low melting point. The tests were then made with Easy-Max. The results are not as expected. Chocolates made without tempering method (which should therefore not be in the form V) and cocoa butter and couverture chocolate (which are in the form V) give the same melting temperature when no temperature ramp is applied. When a temperature gradient is applied, results are difficult to interpret because they present more melting peaks (also for the cocoa butter for example). The Easy-Max is not a suitable instrument for this kind of calorimetric analysis because of the interpretation. The analysis could be performed properly if the DSC could be cooled.

Finally, organoleptic tests were performed on different homemade chocolates and show that it is very important to temper chocolate for taste, texture and optimum brightness.

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### INTRODUCTION

The chocolate is a product which is appreciated and consumed by a lot of people. The main component of chocolate is cocoa butter. The chocolate must have a good taste, texture and an optimal shine. For this, it is important to follow specific temperature ramps; it's called tempering. With tempering, the cocoa butter can crystallize in the form V which is the most stable form.

In this project, two devices were used : DSC and Easy-Max.

### Goal of the project

- Characterize the different polymorphic forms of the cocoa butter by a calorimetric method
- Implement a procedure to manufacture a chocolate with the form V
- Analyse the pure cocoa butter and the chocolate by DSC
- Make chocolate with different tempering methods
- Make chocolate without tempering method to observe other crystalline forms
- Analyse the pure cocoa butter and the chocolate by Easy-Max
- Do organoleptics tests

### Polymorphics forms

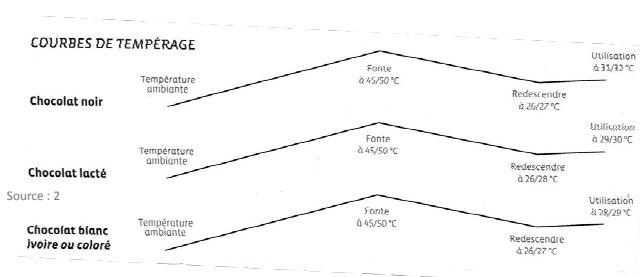
Forms		Melting point [°C]
I	Sub- $\alpha$	17
II	$\alpha$	23
III	$\beta'$	25
IV	$\beta'$	27
V	$\beta$	34
VI	$\beta'$	36

Source : 1

### Advantages of the form V:

- Smooth
- Shiny
- Easy to unmold
- Crisp
- Only melt in the mouth
- Good texture in the mouth and good taste

### Tempering curve

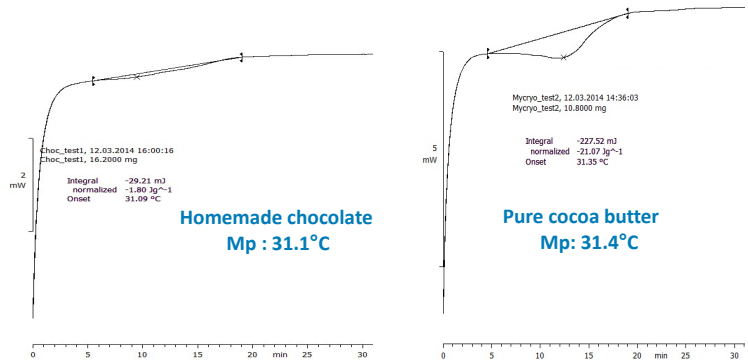


Source : 2

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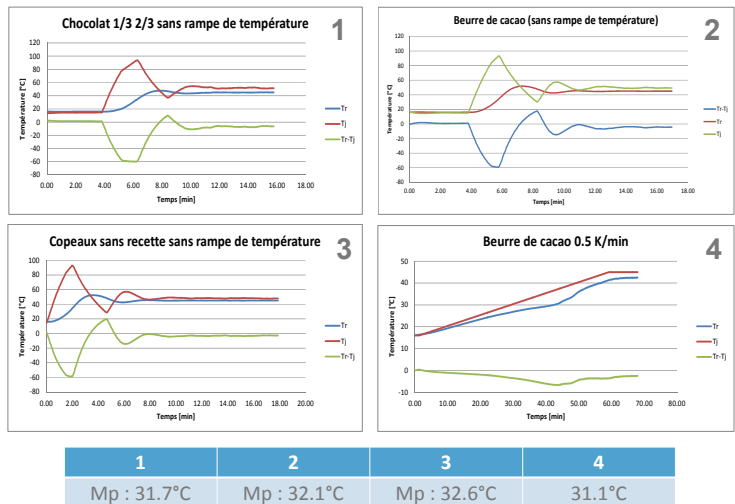
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### DSC results



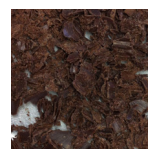
The DSC presents inconclusive results. In fact, it is impossible to cool the DSC apparatus below 25°C, so the chocolate and the cocoa butter are already soft before the analysis.

### Easy-Max results

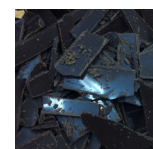


The Easy-Max presents inconclusive results. Chocolates made without tempering method (which should therefore not be in the form V) and cocoa butter and homemade chocolate (which are in the form V) give the same melting temperature when fast temperature ramp is applied. When a temperature gradient is applied, results are difficult to interpret because they present more melting peaks.

### Organoleptics tests



Chocolate without tempering method



Chocolate with tempering method

The pictures show that it is important to temper the chocolate to have all the advantages previously cited.

### CONCLUSIONS

- The DSC must have a cryostat for chocolate analysis
- Easy-Max is not a suitable instrument for this kind of calorimetric analysis
- Tempering is very important to have a good chocolate