ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL OF SCIENCE ENGINEERING AND TECHNOLOGY

DEVELOPMENT OF HEAT RESISTANT COMPOUND CHOCOLATE

M.Sc. THESIS

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Department of Food Engineering

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SEPTEMBER 2016

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İSTANBUL TEKNİK ÜNİVERSİTESİ ★ FEN BİLİMLERİ ENSTİTÜSÜ

ISIYA DAYANIKLI KOKOLİN GELİŞTİRİLMESİ

YÜKSEK LİSANS TEZİ

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EYLÜL 2016

Evindar Doğan, a M.Sc. student of İTU Graduate School of Science Engineering and Technology student ID 506141513, successfully defended the thesis entitled “DEVELOPMENT OF HEAT RESISTANT COMPOUND CHOCOLATE”, which she prepared after fulfilling the requirements specified in the associated legislations, before the jury whose signatures are below.

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FOREWORD

This study is relevant to develop a heat tolerant chocolate, especially for the people who live in warm climate countries. This project has been being worked at an industrial level and I’ve been a part of it during my Erasmus programme.

As I see in my limited time, the heat tolerant chocalate can be produced, even it can resist against higher temperatures than the targeted in our project. Plenty of recipes were prepared and both reasonable cost and non-waxy mouthfeel chocolate was developed.

Since the project is confidential, I shared my outcomes coded. It’s nice to know the results have been being improved in Belgium and I’m proud as a peacock to be a part of the success.

I would like to thank you to Assist. Prof. Dr. Ebru FIRATLIGİL to guide and support me for this master thesis.

September 2016

Evindar DOĞAN
(Food Engineer)

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ABBREVIATIONS

CB	**:** Cocoa Butter
CBE	**:** Cocoa Butter Equivalents
CBR	**:** Cocoa Butter Replacers
CBS	**:** Cocoa Butter Substitutes
CMC	**:** Carboxymethyl Cellulose
DSC	**:** Differential Scan Calorimetry
FA	**:** Fatty Acids
HPKO	**:** Hydrogenated Palm Kernel Oil
HPKOo	**:** Hydrogenated Palm Kernel Olein
HPKS	**:** Hydrogenated Palm Kernel Stearin
HRS	**:** Heat Resistant Chocolate
HS	**:** Heat Stability
Hydroc	**:** Hydrocolloid
MCC	**:** Microcrystalline Cellulose
PE	**:** Polymer Ethylcellulose
PGPR	**:** Polyglycerol Polyricinoleate
PKO	**:** Palm Kernel Oil
PKS	**:** Palm Kernel Stearin
pNMR	**:** Pulsed Nuclear Magnetic Resonance
SFC	**:** Solid Fat Content
STS	**:** Sorbitan Tristearate

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DEVELOPMENT OF HEAT RESISTANT COMPOUND CHOCOLATE

SUMMARY

The presented study investigated hard compound chocolate coating which stays stable in warm climate maintaining a good chocolate taste and mouthfeel. Since chocolate products melt at around 30 °C, consumption of chocolate is unfavorable especially for tropical regions. It is covetable to raise this melting point, in such a way that it remains solid at elevated temperatures. To achieve this, adaptation of the fat composition of the cocoa butter substitutes was studied and checked whether it enabled to increase the heat tolerance of a dark compound coating up to 35 °C or higher.

In addition, the incorporation of a hydrophobic biopolymer created a secondary particle network supporting the structure of compound coatings and contributed to an increased heat tolerance of compound chocolate. Meanwhile, incorporating of a high-melting emulsifier such as mono- and diglycerides supported to increase in heat resistance and non-waxy mouthfeel.

In this research, palm kernel oil and its hydrogenated stearin and olein fractions (HPKS and HPKOo) were applied in a hard compound coating. Several fat mixtures were prepared and thermal anaylsis and solid fat content profiles of the cocoa butter substitutes, showing differences in chemical and functional properties, have been investigated followed by application trials of their respective compound coating on sweet baked goods. Furthermore, screening of hydrocolloids which are alginates that are suspended in fat or in combination with water and sugar added to compound matrix, MCC/CMC/emulsifiers, MCC/CMC/modified starch and non-colloidal MCC *vs*. colloidal MCC studies had been worked.

During experiments, tablet chocolates and various bakery applications, such as donut, cake, muffin, biscuit, wafer were utilised and due to the inequable fat content of applications, a lot of different outcomes were attained. one-week and two-week shelf life testings were carried out and it was proceeded by electing and comparing the best outcomes.

As processing stage, a ball mill refiner was utilised and neither conching nor tempering processes were targeted to attain both glossy appearance as well as time and cost reduction. High shear mixing technique was utilised in some experiments to obtain compound coatings at reduced fat content and to check whether exhibiting similar flow properties with whisk technique or not. Fineness always kept among 18-23 μm after ball mill refining.

ISIYA DAYANIKLI KOKOLİN GELİŞTİRİLMESİ

ÖZET

Çikolata, tropik kakao ağacının tohumlarından elde edilen, içerisinde kakao yağı, kakao tozu, şeker, genellikle süttozu ve çeşitli katkı maddeleri bulunduran, kodeks standartlarına dayalı üretilen ve kalıplanarak son şekli verilen bir gıda ürünüdür. İçerisindeki katı taneciklerin genellikle kakao yağı içeren bir sıvı matris içerisinde dağılmış olarak bulunduğu bir süspansiyon olarak tanımlanır. Çikolata esaslı veya kaplamalı gıda ürünlerinin yüksek miktardaki talebinden dolayı uzun yıllardır dünya çapında en çok tercih edilen gıda çeşidi ve aromasıdır. Ancak gerçek çikolata 33.8 °C de tamamen eriyen bir yapıya sahip olduğundan tropikal ülkeler için tüketimi oldukça elverişsiz olduğundan geliştirilmesi arzu edilmektedir.

Bu çalışmanın amacı, fırıncılık ve pastacılık ürünlerinin kaplaması amacıyla kullanılmayı hedefleyen ve 35 °C de yapısını ve şeklini koruyacak ısıya dayanıklı kokolin geliştirmektir. Üretilecek ürün mumsu, yapışkan tatta olmamalı, temperleme işlemsiz tüketime elverişli olmalı ve kaplama için gerekli akış özelliklerine sahip olmalıdır. Bu kokolin, içeriğinde kakao yağı yerine bitkisel yağlar içerdiğinden gerçek bir çikolata değil, çikolata türevi bir üründür. Maliyeti azaltma, ısıya dayanım verme ve maksimum ölçüde gerçek çikolata tadı, görünüşü ve yapısını aratmama hedefleri ile tasarlanmıştır. Kokolin, konçlama veya temperleme işlemlerine ihtiyaç duymaz; tat olarak ilk etapta gerçek çikolatadan kolayca ayırt edilir.

Çikolata üretimi kakao çekirdeğinden itibaren çok çeşitli proseslere maruz kaldığından karışık bir süreçtir ve süre, sıcaklık, hammadde kalitesi ve üretim parametreleri gibi çok sayıda faktör çikolata üretimini etkiler. Fakat dünya çapında yürürlükte olan gıda kanun ve yönetmeliklerine dayanarak söylenebilir ki, çikolata çeşidi içerdiği hammaddeye göre isimlendirilir ve kakao kitlesi ilavesi ve/veya süt içerimine bakılarak bitter, sütlü veya beyaz çikolata olduğuna karar verilir.

Gerçek çikolata yapımında üç çikolata çeşidi için de temel işlemler uygulanır. En belirleyici üretim aşamaları hammadde karışımı, inceltme, konçlama, viskozite ayarlama ve temperlemedir. İşleme öncesi bitter ve sütlü çikolatalar için hem kakao tozu hem de sıvı formda saf kakao kitlesi içeren kakao likörü alkalizasyon işlemine tabi tutulur. Beyaz çikolata, kakao türevlerinden sadece kakao yağı içerdiğinden alkalizasyona ihtiyaç duymamaktadır. Kokolin yapımında, hammaddeler ilk olarak toz halde karıştırılır ve pürüzsüz bir yüzey elde etmek için inceltilir. Daha ucuz bitkisel yağlar ve kakao tozu sırasıyla kakao yağı ve kakao likörünün yerine kullanıldığından konçlama ya da temperleme işlemleri olmadan da çikolata üretilebilir.

Zaman-sıcaklık kombinasyonuna dayanan hammadde karışımı çikolatada ilk üretim prosesidir. Sabit kıvam elde etmek için, sürekli veya kesintili çalışan karıştırıcılardan yararlanılır. Kesintili karıştırmada, hammaddeler 12-15 dakika arası çikolata çeşidine bağlı olarak 40–50 °C arasında karıştırılır. Sürekli karıştırma yöntemi büyük ölçekli çikolata fabrikaları tarafından tercih edilir ve sert bir yüzey ve plastik kıvam üretmek

için iyi bilinen otomatik yoğuruculardan geçirilir. Modern çikolata endüstrisinde istenen pürüzsüz çikolata yüzeyini elde etmek için, inceltme en önemli basamaktır. Çikolata çeşidine bağlı olarak, gerçek çikolata beş silindirli inceltici ile inceltilir; kokolinde ise 30 μm den az tanecik büyüklüğü elde etmek için bilyeli öğütücüler kullanılır. Konçlama, çikolatanın aroması, vizkozitesi ve son yüzeyini geliştiren en temel üretim basamağıdır. 50 °C den yüksek sıcaklıkta birkaç saat çikolata sarsılarak karıştırılır. Çikolatanın çeşidine bağlı olarak değişen süre ve sıcaklıkta ilk aşamada nem azalır, konçlamanın sonlarına doğru ise ekstra kakao yağı ve emülsifiye edici maddeler uygun vizkozite değerine ulaşmak için ilave edilir. Temperleme parlak görünüş sağlamak ve istenmeyen beyaz yağ çiçeklenmelerini engellemek için olmazsa olmazdır. Ayrıca, iyi temperlenmiş çikolata güzel şekilli, stabil, daha sert ve daha ısı dayanımlıdır.

Bitter çikolata kakao katı maddeleri ile şeker parçacıklarının kakao yağının kesintisiz fazı içinde bir süspansiyonudur. Bu faz 25-36% arasında değişen katı yüzdesine sahiptir ve 32 °C civarlarında erir. Reolojik çerçevede bakıldığında, çikolatanın en etkileyici durumlarından biri, çok küçük değişikliklerin akış özelliklerinde çarpıcı değişiklikler yaratabilmesidir. Çikolata çok parametreli ve zor bir üretim sürecine ihtiyaç duyan, birçok değişkeninin reolojik, duyusal ve fonksiyonel olarak sürekli kontrol altında tutulması gereken bir gıda ürünüdür.

Kokolinlerin tercih görmesinin nedenleri arasında kakao yağının düşük süt toleransı, yüksek sıcaklıklardaki stabilizasyon eksikliği ve yağ çiçeklenmesi eğilimidir. Ayrıca, kakao yağının ekonomik ve teknolojik durumları göz önüne alındığında, alternatiflerinin arayışına başlanmıştır. Kakao yağı yerine geçen bitkisel alternatifler laurik asitten elde edilen ve edilmeyen diye ikiye ayrılır. Hindistancevizi yağı ve palm çekirdeği yağı (PKO) laurik yağ kaynaklarıdır ve kakao yağıyla bazı uyuşmazlıklar gösterse de benzer fiziksel özelliklere sahiptir.

Laurik alternatif yağlar stearin yada olein gibi fraksiyonlarına ayrıldığında, daha güzel yüzeye, stabiliteye ve aroma açığa çıkışına sahip olurlar. Ek olarak, kısa zincirli yağ asitlerince zengin olan bu yağlar maliyetçe daha düşüktür ve temperlenme gerektirmezler. Palm çekirdeği stearinleri (PKS) kakao yağından kimyasal olarak tamamen farklıdır ve sadece fiziksel olarak bazı yüzey benzerlikleri vardır. Gevreklik ve başlangıçtaki parlaklığı sağladığı için iyi kalitede kaplama sonucu verir. Ayrıca, iyi oksidatif stabilite, temperlenmeden hızlı katılaşma ve kakao yağından daha uzun raf ömrü avantajlarına sahiptir. Ancak, kaplama yönünden kakao yağıyla uyuşmazlık ve neme maruz kaldığında sabunsu kötü tada sahip olma gibi dezavantajları mevcuttur.

Bir diğer kaplama amaçlı kakao yağı alternatifi de hidrojenize edilen laurik yağlardır. Hem ucuz olması hem de kullanım kolaylığı açısından en çok tercih edilen kaynak hidrojenize palm çekirdeği yağıdır (HPKO). Ürünü hidrojence zenginleştirme katılaşmasını sağlar. Önce fraksiyonlarına ayırıp stearin veya olein formunu alan oldukça güçlü bir kakao yağı alternatifi olan PKO daha sonra hidrojenize edilerek iki kat daha etkili bir form istenirse alabilir. Düşük katı yağ içerikli palm çekirdeği oleini hidrojenize de edilirse katı yağ içeriği (SFC) yükseltilmiş olur. Hidrojenize yağlar arasında en keskin erime profilini sergileyen yağ hidrojenize palm çekirneği stearinidir (HPKS), onu da HPKO takip eder. Hidrojenize palm çekirdeği oleini (HPKOo) de bu iki yağ çeşidine göre düşük sıcaklıklarda daha yumuşak bir eğilim gösterme içindedir.

Bu projede bütün faktörler göz önüne alınarak ilerde kaplama amaçlı endüstriyel düzeyde kullanılan ve tropik ülkelere satışı yapılan ısıya dayanıklı bitter kokolin çalışmaları yapılmıştır ve kakao yağı yerine geçen bitkisel alternatif yağ çeşitleri denenmiştir. PKO ve onun yukarda bahsedilen hidrojenize stearin ve olein fraksiyonları deneylerde kullanılmıştır. Yağ bileşimlerinin adaptasyonu üzerine yoğunlaşılmış ve 35 °C de ki ısı dayanımı incelenmiştir. Birbirlerinden kimyasal ve fonksiyonel özellikleri farklı çeşitli yağ karışımlarının termal analizleri yapılmış ve SFC profilleri çıkarılmıştır. Ayrıca çeşitli hidrokolloidler eklenerek de denemeler yapılmış, yağda askıda kalma veya su ve şekerle kombine edilerek ekleme metodları uygulanmıştır. Mikrokristalin selüloz ve/veya karboksimetil selüloz gibi hidrokolloidlere ayrıca çeşitli emülgatörler eklenerek de çalışmalara devam edilmiştir. Emülsifiye edici özelliği olan lesitinlerden yararlanılmış, akma geriliminin ve erimiş çikolatanın vizkozitesinin azaltılması sağlanmıştır. Lesitinlerin rolü su emici şeker taneciklerini topaklanmadan limitlemek ve istenen akışkanlığa destek olmaktır. Çalışmalar nişasta denemeleriyle sonlanmış ve tüm deney sonuçlarına göre en elverişli, uygun fiyatlı, yapısını ve tadını bozmayan hammaddelere karar verilmiştir.

Deneyler süresince, tablet çikolata şeklinde kokolinler üretilmiş ve donut, kek, muffin, bisküvi, gofret gibi çeşitli pastacılık ürün uygulamaları kaplanmıştır. Bu ürünlerin başlangıç yağ miktarları birbirinden farklı olduğundan sonuçların farklılık gösterdiği saptanmıştır. Bir ve iki haftalık raf ömrü çalışmaları yapılmış ve sonuçlara başarısı dahil edilmiştir.

Proje boyunca üretilen kokolinlerin üretim sürecinde, inceltme işlemi için bilyeli öğütücü kullanılmış ve kokolin üretimi sadece hammadde karıştırma ve inceltme süreçlerinden geçmiştir; konçlama ve temperleme uygulanmamıştır. Parlak görünüm, zaman ve maliyet kazancı hedefleri tek seferde elde edilmeye çalışılmıştır. Üretim yapılırken hem klasik çırpıcılardan hem de yüksek hızlı karıştırıcılardan yararlanılmış, benzer akış özellikleri gösterip göstermedikleri ve sonuca etkileri gözlenmiştir. Öğütme inceliği kontrolleri sürekli her inceltme işleminden sonra kontrol edilmiş, 18-23 µm arasında kaldığından emin olunmuştur.

Deneyler sonunda 35 °C de tüm hedefleri sağlayan ısıya dayanımlı kokolin geliştirilmiş, ve proje başarıya ulaşmıştır. Çalışmalar Belcolade, Puratos Group, Belçika da tamamlanmış olup endüstriyel düzeyde daha başarılı ve kullanıma elverişli, daha ekonomik, daha yüksek ısıya dayanıklı çikolata veya çikolata türevi ürün hedefi ile yeni hipotezlerle yeni bir proje üzerine yoğunlaşılırsa hedeflerin gerçekleşeceği beklentimdir.

1. INTRODUCTION

Chocolate is identified as a suspension of solid particles that are dispersed within a continuous lipid matrix consisting of both solid and liquid fat, which is mainly cocoa butter (CB) (De Graef et al., 2011). It is one of the most preferred food types and flavors worldwide since a great quantity of foodstuffs which can be either chocolate-based or -coated, have been produced in many years.

Chocolate can be evaluated as a friend and it is accepted as a functional food in recent years. Contrary to what is belief, CB does not raise level of cholesterol. Modern studies reveal that, neutral impacts of stearic acid found on blood cholesterol hinder to raise. Besides, chocolate have a dominant role in supplying some nutrient clusters. Essential fatty acids (FA) balance triglycerides nutricionally as well as natural antioxidants found in chocolate enhance in vitro and in vivo conservation of lipids. Furthermore, dark chocolate retain nutritional value of cocoa since it has a advanced proportion of cocoa than milk chocolate (Shukla, 2006).

The process of chocolate manufacturing is complex since it requires several processing steps starting from cocoa beans. Besides, various factors influence its production, containing the selection of components, process parameters, time and temperature. However, if it is to be used in large industrial applications or not, the basic manufacturing process remains the same. In accordance with several food law and legislations throughout the world, there are certain kinds of ingredients which are involved in chocolate type identification. Dark, milk and white chocolate recipes are distiguished from each other at the point of cocoa mass inclusion as well as milk content inclusion. However, United States circumstances are stricker than European Union and Codex Standards, in terms of compositional structure, labelling requirements and restricted amounts of ingredients (Yates and Callebaut, n.d.).

Since chocolate completely melts at 33.8 °C, consumption of it is unfavorable especially for tropical regions. It is covetable to raise this melting point, in such a way that it remains solid at raised temperatures (Stortz and Marangoni, 2011).

1.1 Purpose of Project

The objective of this project is development of heat resistant chocolate (HRC) which targets a hard compound coating for bakery and patisserie products and able to withstand elevated temperatures up to 35 °C, *i.e.* isn't sticky and keeps its shape. Furthermore, this innovation must exhibit no waxy mouthfeel. Additionally, it should answer the flow requirements for enrobing and needs to be applicable without the necessity to temper.

1.2 Literature Review

1.2.1 Real *versus* compound chocolate

1.2.1.1 Composition

Cocoa mass, milk solids and fat, sugar or other permitted ingredients change according to be milk, white or dark chocolate recipes. Even though ingredient amounts vary depending on the types, all chocolate recipes comprising white chocolate need to include CB (Afoakwa, 2014a). When the chocolate shows incompatibility with the standards of legislations, it must be referred to as compound coating which is also called as confectionary coating (Yates and Callebaut, n.d.).

Compound chocolate is a chocolate-derived product involving vegetable fats other than CB. Generally compound chocolate is utilised for coatings of food products owing to its lower cost (Toker et al., 2015). Real chocolate and compound chocolate are commonly produced in a different way. In fact, compound chocolate does not require conching and tempering processes, and so different applications are more feasible to compound coating since it has more all-purpose features and is convenient in terms of fortification, coloring or flavoring. Due to the differences in processing, compound and real chocolate can be distinguished easily by tasting (Aebi, 2009).

1.2.1.2 Processing

The production of chocolate includes several chemical and physical processes which require various technological operations. To obtain the desired quality attributes, different ingredients should be added during processing (Afoakwa, 2014a). Figure 1.1 shows the model of chocolate including its phases and main ingredients.

In real chocolate, basic operations are carried out to dark, milk and white chocolate, more particularly ingredient mixing, refining, conching, standardization of viscosity and tempering. Both cocoa powders and cocoa liquor which is pure cocoa mass in liquid form, are exposed to alkalization as prior processing for dark and milk chocolate because of white chocolate involves CB only (Tanabe and Hofberger, 2005).

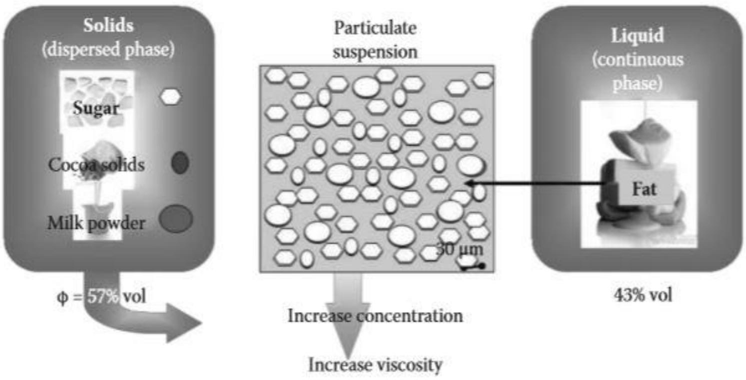


Figure 1.1 : Chocolate model (Afoakwa, 2014a).

For compound chocolate, the ingredients are dry mixed and refined to get a smooth texture using a ball mill refiner. Since cocoa butter and cocoa liquor have been replaced with respectively less expensive vegetable fat and cocoa powder, compound chocolate does not need both conching and tempering processes. Processing steps are charted for real milk chocolate in Figure 1.2 (Tanabe and Hofberger, 2005).

Mixing

The first operation in chocolate manufacturing is ingredient mixing according to time–temperature combinations. To attain constant formulation consistency, continuous or batch mixers are utilized. In batch mixing, ingredients are mixed for 12 to 15 min at 40–50 °C depending on chocolate type. In continuous mixing, well-known automated kneaders are preferred by large chocolate manufacturers to produce quite tough textures and plastic consistencies (Afoakwa, 2014a).

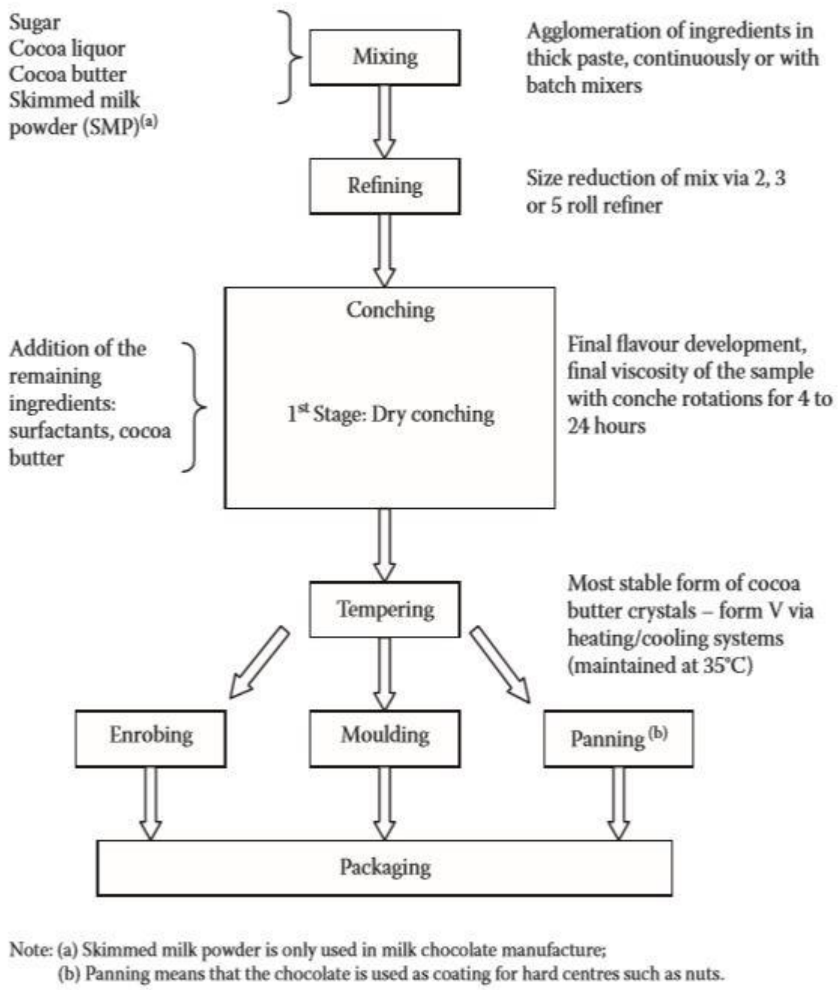


Figure 1.2 : Real milk chocolate production flow chart (Afoakwa, 2014a).

Refining

To attain a smooth texture which is desired in modern chocolate confectionery, chocolate refining is a significant step. Depending on the chocolate type, ingredient mixtures is refined by five-roll refiners for real chocolate and ball mills for compound coating to get particle size <30 µm for 15 min (Afoakwa, 2014a).

Conching

Conching is considered as the endpoint in the bulk chocolate production, independently of the chocolate type. It is a fundamental process which makes contribution to improvement of flavor, viscosity and final texture. Normally it is implemented by agitating the chocolate at >50 °C for a few hours. In first stage, reduction of moisture occurs. Conching times and temperatures vary depending on the chocolate type and desired flavor as well. In second stage, extra CB and lecithin is added towards the end of the process to obtain proper viscosity values (Afoakwa, 2014a).

Tempering

Six polymorphic forms indicated by Roman number (I–VI) or in Greek letters (γ, α, β'_2, β'_1, β_2, β_1) were found CB. Form V is the most desired form in chocolate, which ensures a glossy appearance, contraction from the mould, and resistance to fat bloom (Talbot, 2009a). In addition, following features are the consequences of a well-tempered chocolate: good shape, stable, harder, more HRC product (Afoakwa, 2014a).

Four key steps are identified in tempering. The first stage is melting to completion at 50 °C, the second one is cooling to point of crystallisation of about 32 °C depending on chocolate type, the third one is crystallization of about 27 °C depending on chocolate type and the last one is conversion of any unstable crystals at 29–31 °C (Afoakwa, 2014a)

From the first in processing chocolate, cocoa beans are cleaned after exposed to harvesting, fermentations and sun drying. Then, they are subjected to roasting and winnowing processes to enhance aromatic characteristics of the bean and remove the shells from nibs respectively. Manufacturing steps are explained in detail as follows: The cocoa nibs are pre-ground to reduce the particle size of cocoa solids. The goal is to adapt settings of pre-refiner to obtain a good and workable mass for next step. Industrial grinding is performed in several steps in order to achieve the correct fineness, mostly using five-roll refiner. For compound chocolates a ball mill refiner is preferred. For milk chocolate, resulting chocolate liquor is mixed with extra CB, sugar and milk. The final mixture should include almost 30% fat, 50% sugar and 20% cocoa powder. For white chocolate, cocoa solids are replaced with milk powder and non-cocoa compounds can be comprised except CB. Besides, for dark chocolate, if only cocoa powder is used into mixture instead of cocoa liquor, the manufacturing does not need the roasting, winnowing, pre-refining and grinding processes. Conches are then utilised to attain the real chocolate in combination with moisture control and temperature. Conching process changes rheology of chocolate, removes moisture and volatiles and develops mouthfeel. Emulsifiers like lectihin and flavors can be added on the the chocolate mixture during conching. Before tempering, a low viscosity is targeted. In a final step, tempering is implemented which is controlled cooling of melted real chocolate with agitation. In compound chocolate manufacturing, ball mill refiner is the last step of CB alternatives with mixtures of cocoa powder and sugar (Beckett, 2008; Tanabe and Hofberger, 2006; Hofberger and Tanabe, 2007).

1.2.1.3 Quality

In chocolate manufacturing, the crystallization of CB and chocolate composition have a significant role attaining a high quality product. CB crystallization is the most important factor in real chocolate manufacturing and processing. If chocolate crytallizes inadequate, formation of fat bloom is seen. Besides, different interactions are determined by chocolate composition, which take place among ingredients (Afoakwa et al., 2009a). Rheological measurements of viscous liquid and sensory evaluation of final solid product control the chocolate quality (Afoakwa, 2014b).

Flow properties

Rheologically, molten chocolate acts as a non-Newtonian liquid and reveals as non-ideal plastic behavior. Its flow behavior can be expressed by both a yield stress which is the minimum stress to initiate flow and a plastic viscosity related to the internal friction against flow (Fernandes et al., 2013).

Chocolate flow is influenced by the processing stages, such as refining, conching and tempering as well as by composition, such as fat type and content, amount of emulsifiers and particle size distribution which is affected by ratio of solid ingredients but also by refining conditions (Vavreck, 2004; Schantz and Rohm, 2005; Afoakwa et al., 2009b).

Additionally, time-dependent behavior is exhibited by chocolate; in other words, a change of viscosity and shear stress at a given shear rate happens with time. This phenomenon is related to change in the material structure. Thixotropy can be excountered because of decreasing of viscosity with time of shearing, when the stress is removed followed by recovery of the structure. In case of real chocolate, a well-conched chocolate should not be thixotropic (Servais et al., 2004; Afoakwa et al., 2008b).

Dark chocolate is a suspension of cocoa solids and sugar particles, in a continuous phase of CB (Beckett, 2009). The CB phase has a mass percentage changing between 25-36% and melts when heated above almost 32 °C. From a rheological framework, one of the most impressive aspects of chocolate is that minor changes in chocolate composition can strikingly change the flow attributes (Afoakwa et al., 2008a).

In chocolate industry, lecithin is an emulsifier which decreases the yield stress and viscosity of molten chocolate (Garti and Aserin, 2012). Its role is to limit the hydrophilic sugar particules from aggregating and help the flow by lubrication (Afoakwa et al., 2008a). Also, polyglycerol polyricinoleate (PGPR) which is a hydrophobic emulsifier is commonly utilised in chocolate manufacturing since it makes the chocolate flow easily. It shows a synergy with lecithin by enhancing the chocolate texture and raising the fat crystallization rate (Garti and Aserin, 2012).

Sensorial and functional properties

Some distinct sets of sensory characteristics are present at chocolate, that are texture appearance and flavor. They have a significant role on acceptability and preferance by consumers. The most important sensory characteristic of chocolate is flavor, which is affected by texture, taste and aroma during consumption (Afoakwa, 2014b).

Furthermore, when comparison among the CB-based chocolate and the compound coating fat chocolate, varying differences in sensorial and functional properties are encountered. Some of these differences are owing to their melting profiles and others are sourced more by differences in crystallisation and post-crystallisation (Talbot, 2009b).

Melting profile

Typical melting profiles of the several compound coating fat, identified by solid fat content (SFC) are shown in Table 1.1 as well as several sensory features, such as waxiness, coolness and hardness can be defined by the melting profile, are demonstrated in Figure 1.3 (Talbot, 2009b).

Table 1.1 : SFC[a] of compound coating fats (Talbot, 2009b).

SFC	CB[b] (%)	CBE (%)	CBR (%)	CBS (%)
N20	76.4	74	88	82
N25	67.2	64	71	67
N30	41.5	47	40	28
N35	0.4	3	6	1
N40	0.1	0	0	0

[a]SFC of CB and CBE measured following IUPAC method 2.150(b); SFC on CBR and CBS measured following IUPAC method 2.150(a). See IUPAC Standard Methods fort he Analysis of Oils, Fats and Derivatives, 8th edition, 2.150 Solid content determination in fats by NMR. [b]Ghana CB (from Wong Soon, 1991).

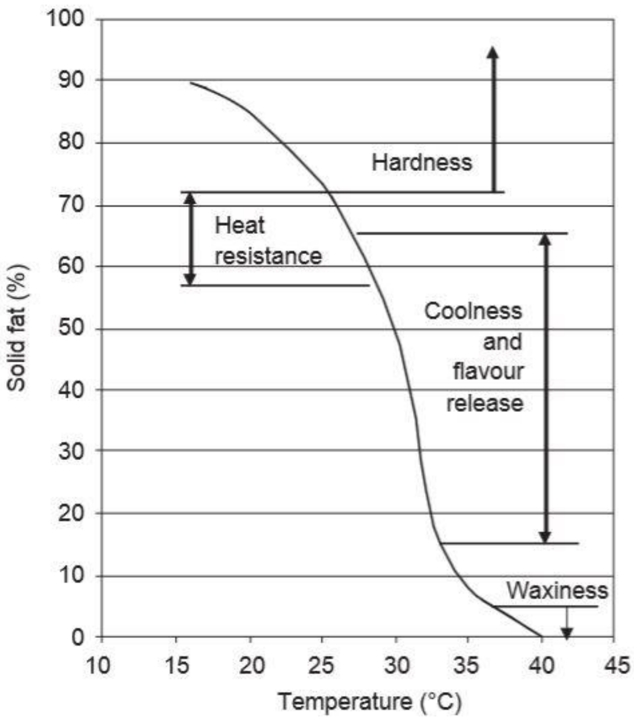


Figure 1.3 : Melting profile of confectionery fats (Talbot, 2009b).

A higher brittleness in coatings is the consequence of higher SFC at temperatures of 15–25 °C. CBR and CBS have the highest SFC at 20 °C. They also generate coatings which have a pronounced snap when they are biten. The heat resistance of a compound fat rises with temperature, particularly at 25-30 °C, if more solid fat is present. Especially in the climates with higher ambient temperatures, this behavior makes this type of coating more convenient (Talbot, 2009b).

Even at 40 °C, modified CBR/CBS still can have a substantial amount of solid fat. Above 37 °C, any fat residuary in the solid form will be perceived as waxy. This means that a balance has to be obtained among minimal waxiness and heat resistance (Talbot, 2009b).

Even though in Table 1.1, it would be seen that the non-lauric CBR would exhibit minimal waxiness, these kinds of compound coating fat can demonstrate post-hardening on storage. Generally, post-hardening is a consequence of a further, slow crystallization after production and packing as well as around ambient temperatures, it in chocolate results in an increase in the SFC. Hereby, improvement of the brittleness of the chocolate is encountered. Besides, post-hardening in non-lauric CBR can often result in a increase of SFC at 35 °C that gives a waxier coating (Talbot, 2009b).

1.2.2 Confectionery fats

1.2.2.1 Cocoa butter

The main confectionery fat which is mainly composed of three FA, namely palmitic, stearic and oleic acid, is considered as cocoa butter. It also has a very simple triglyceride composition which is predominated by POP, POSt and StOSt (P = palmitic, St = stearic, O = oleic) (Talbot, 2009c).

These triglycerides are responsible for supplying the specific CB crystallization and melting characteristics. Particularly, the sharp melting behavior supplies a cooling effect in the mouth when consuming the chocolate. Hereto, CB substitution is vital in various respects. Firstly, the melting behavior has to be similar to with CB in order to attain the same mouth feel. Secondly, the addition of the fat must not change severely the melting behavior and crystallisation of CB if it is only partially substituted (Lipp and Anklam, 1998).

Also fractionation can be carried out to CB. It is stated as a process in which higher melting triglycerides are distinguished from lower melting triglycerides. According to Weyland (1992) “the higher melting (SOS-rich) triglycerides concentrate more into the stearine fraction while the lower melting (SOO- and SLiS-rich) triglycerides concentrate more into the oleine fraction” (Weyland, 1992). This stearine fraction can be utilised in chocolate which needs a higher degree of heat tolerance (Talbot, 2009c).

1.2.2.2 Cocoa butter alternatives in compound coatings

CB has some drawbacks such as low milk tolerance, lack of stabilization at raised temperatures and fat bloom tendency (Shukla, 2006). In addition, due to economical and technological situations of CB, its alternatives have been set off on a quest. (Lipp and Anklam, 1998).

In accordance with researches, there is no other naturally occurring fat equal to CB in terms of physical properties, since at body temperature, it is fast and completely melting as well as at room temperature it is brittle. Thus, all alternatives are obtained by fat modification and/or blending. And the major methods to modify the fats are interesterification and fractionated crystallisation of it along with breeding the plants as selectively (Lipp and Anklam, 1998).

Vegetable fats that are substituted for CB can be classified as lauric and nonlauric. Coconut oil and palm kernel oil (PKO) are sources of lauric fats which have similar physical attributes but show incompatibility with CB. When coconut and PKO are fractionated, they will have good texture, stability and flavor release. Lauric fats which are rich in short-chain FA do not need tempering and they are cheaper than the CB (Minifie, 1989).

Non-lauric fats which contain hydrogenated oils such as palm, soy and cottonseed are comprised of longer chain FA. They can be blended with cocoa liquor and up to 25% CB to achieve a stronger flavor. Since their texture is quite different and less brittle from CB, they are often utilized as cover for baked products. Both lauric and nonlauric fats are sensitive to decomposition by lipase enzyme. While lauric has a soapy flavor when it has been exposed to lipase, FA of non-lauric fats do not (Minifie, 1989).

In compound coating, there are three main kinds of fat used. The first category is cocoa butter equivalents (CBE), blends of vegetable fats or fractions which are similar to CB in terms of triglyceride composition. The second one is non-lauric cocoa butter replacers (CBR) which are based on fractionated and/or hydrogenated oils. The third category is lauric cocoa butter substitutes (CBS) that can be based on fractionated and/or hydrogenated oils as well. Preferance of one coating over another is done according to three main aspects that are processing capabilities, flavor and nutritional requirements (Talbot, 2009c). The attributes of three fat categories are demonstrated in Table 1.2 in detail.

Table 1.2 : CB alternatives examples, properties and composition (Brinkman, 1992).

Cocoa alternative	Plant fat type	Function	Main FA	Main triglycerides
CBE	Palm oil, illipe butter, shea butter, kokum butter, sal fat	Non-lauric fat, does not alter the properties of CB in mixtures	Palmitic, stearic oleic, linoleic arachidic acid	POP, POS, SOS
CBR	Soya oil, rape seed oil, cotton oil, palm olein	Partially compatible different triglycerides	Elaidic acid, stearic acid, palmitic, linoleic	PEE, SEE
CBS	Coconut oil, PKO, Medium chain triglycerides	Lauric fats, suitable for 100% substitution only	Lauric, myristic	LLL, LLM, LMM

1.2.2.3 Cocoa butter equivalents

CBE are quite similar to CB in terms of both physical and chemical features. Three main triglycerides such as POP, POSt and StOSt that are also found in CB are included in CBE and they can be mixable with CB in any amount without changing its attributes (Lipp and Anklam, 1998; Talbot, 2009c). The principal advantages of CBE are cost reduction against real chocolate, development of milk fat tolerance, resistance against high temperatures during storage and fat bloom control (Shukla, 2006).

Palm oil, palm-mid fractions, illipe fat, shea butter, sal fat, kokum butter, mango kernel fat and some commercially present mixtures of vegetable fats are sources of CBE (Lipp and Anklam, 1998). Since no single vegetable fat includes all three triglycerides in the same proportions to CB, they need to be blended to produce CBE (Talbot, 2009c). Triglyceride distribution of CBE and CB is indicated in Figure 1.4.

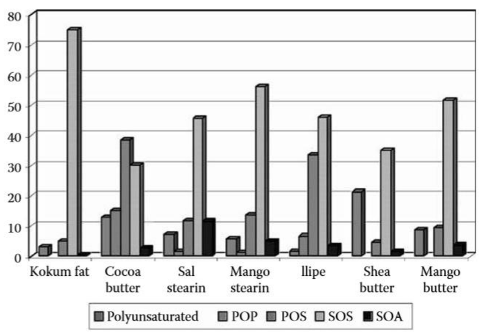


Figure 1.4 : Triglyceride distribution (in wt%) of CBE and CB (Shukla, 2006).

1.2.2.4 Cocoa butter replacers

CBR originate from commodity oils such as rapeseed oil, palm oil, cottonseed oil and soybean oil. Their SFC is fairly low since all of these oils are found in their native form as either liquid or semi-liquid which makes them most inconvenient for use in coatings. Therefore, a combination of hydrogenation and fractionation is carried out (Talbot, 2009b).

After hydrogenation, CBR are fractionated to attain a proper melting profile for compound coatings. Their merits contain no need for the tempering process, heat resistance and high oxidative stability for compound chocolate. However, compatibility of fat with CB is quite lower than CBE. CBR compatibility is only 25% at a max of the total fat content, though CBE is 100%. Also, chocolates made by CBR are of poorer quality compared with CBE chocolates (Yamada et al., 2005). However, it has some more important advantages such as fairly low and stable price, both high (%20) and low (%12) fat content oils and no risk of soapy flavor as well (Shukla, 2006).

1.2.2.5 Cocoa butter substitutes

CBS based on palm kernel stearin (PKS) are totally different from CB chemically and only have some physical similarities (Lipp and Anklam, 1998). They give good quality coating since they ensure brittleness and a good initial gloss (Talbot, 2009b).

Besides, they have some principal advantages such as good oxidative stability, texture similarity with CB, fast solidification without tempering and longer shelf life than CB. However, they have two main drawbacks for coatings, which are incompatibility with CB and risk of soapy off-flavor when they have been exposed to moisture (Young, 1983).

In some cases, the olein fraction of PKO can be recombined with the oil before hydrogenation to produce a fat favorable for lauric usage as CBS. Conversely, the stearin fraction of PKO can be utilised in lauric coatings directly (Talbot, 2009b). The triglyceride and FA composition of commercially available CBS have been studied (Nesaretman and Kun, 1990). No remarkable differences among the fats were specified. They arise from almost 50% lauric acid and 20% myristic acid (Lipp and Anklam, 1998).

Within the framework of the chemical and physical features of CBS; the FA composition and SFC of two commercial CBS are shown in Table 1.3 and Figure 1.5. The trans content of hydrogenated palm kernel stearin (HPKS) is zero (<1%), and it has great mouth feel and heat resistance (Yamada et al., 2005).

Table 1.3 : Major FA composition of typical CBS (Yamada et al., 2005).

Major FA	Lauric (%)	Myristic (%)	Oleic (%)
PKS	55	22	7
HPKS	55	22	0.5

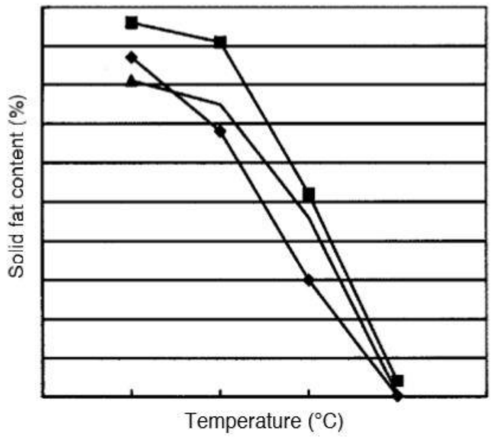


Figure 1.5 : SFC of typical CBS. ■, HPKS; ◆, PKS; ▲, Malaysian CB (Yamada et al., 2005).

Within the framework of production process and attributes of CBS, the PKO, is hydrogenated and/or fractioned. The hard fraction of PKO is known as palm kernel stearin, and to increase heat resistance it is hydrogenated (Yamada et al., 2005).

SFC of PKO is a high at low temperatures but the lowest one is 25 °C. Fractionation process generates further a stearin where the SFC is enhanced at low temperatures. This demonstrates complete melting at 35 °C (Siew, 2001).

Another alternative to chocolate in use a compound coating is hydrogenated palm kernel oil (HPKO) which is seen preferable because both being cheap and also easy to use as it does not need tempering in a controlled manner. Its disadvantages are being imcompatible with CB and having a relatively short shelf life owing to the stability of lauric fat as well. To give an enhanced shelf life, fractionated PKO can be used in place of HPKO, even though the incompatibility with CB is still present (Talbot, 2009b). SFC% of several CBS is shown in Table 1.4.

Table 1.4 : SFC% of PKO products (Siew, 2001).

	10(°C)	15(°C)	20(°C)	25(°C)	30(°C)	35(°C)	40(°C)
PKO	69.4	57.1	36.0	12.3	-	-	-
PKOo	54.8	36.8	17.2	-	-	-	-
PKS	91.8	89.6	83.1	67.1	30.8	-	-
HPKO	94.6	93.6	87.6	64.6	34.9	13.0	7.2
HPKOo	93.6	90.1	79.4	59.6	38.9	19.9	0.6
HPKS	95.4	94.9	94.1	84.8	38.9	4.2	0.5

So, PKO is highly valued for confectionery industry. The low SFC of PKOo can be increased by hydrogenation. Between the hydrogenated products, the sharpest melting profile is demonstrated by HPKS, followed by HPKO. HPKOo is in tendency to be softer at low temperatures (Siew, 2001).

Table 1.5 gives the data of the DSC analyses for PKO products. When PKO is cooled to sub ambient temperatures at steps of 5 °C/min, it crystallises quickly at temperatures relatively below its melting point (Siew, 2001).

Table 1.5 : DSC analysis of PKO products (Siew, 2001).

Sample	Melting program		Crystallisation program
	T_{offset} [°C]	ΔH [J/g]	T_{cry} [°C]
PKO	30.9	100.6	2.8, 5.6
PKOo	27.6	82.1	2.9, 2.2
PKS	36.5	106.6	10.7
HPKO	49.5	120.3	18.9, 27.7
HPKOo	50.6	120.7	20.1, 29.7
HPKS	42.5	106.7	11.8

The merits of CBS include the followings as well. Elimination of transfat, low cost compared with CB, excellent mouth feel compared with CBR (Yamada et al., 2005).

In confectionery industry, modified CBS from hydrogenation and/or fractionation of PKO are mostly used (Calliauw et al., 2005; Lipp and Anklam, 1998; Young, 1983). They are recognized to crystallize directly within β' crystal polymorph and to exhibit similar melting behavior with CB (Garti and Widlak, 2012). HPKS is one of the CBS which plays a significant role in confectionery fats (Wang et al., 2011).

1.2.3 Heat resistant chocolate

Chocolate is a food product which comprises a smooth texture, a shiny gloss and crackle when it is broken. However, there is a big problem with chocolate in summer season and in tropical areas as well. CB melts at 33.8 °C, so researches were centred upon the development of HRC, especially for tropical countries (DeMan, 1999).

A HRC can resist deformation or melting against temperature above 34 °C which is the normal melting temperature of real chocolate (Stortz et al., 2014). Up to now, three main strategies to produce HRC were revealed, namely enhancement of network microstructure, addition of oil/fat binding polymer and raising the fat melting point. Regarding enhancement of the network microstructure, induction of a second sugar network is targeted the incorporation of water, both direct and indirect (Stortz and Marangoni, 2011).

Almost 65 years ago, the first studies were carried out and 4-20% of water was added onto chocolate before heating at above 65.5 °C, so that most of sugar found in chocolate was dissolved with water. The mentioned chocolate was able to withstand against warm climatic conditions. Furthermore, he stated that the chocolate had a final water content of 4-10% and a fat:sugar ratio is greater than 5:7. In conclusion, the product produced had a high viscosity making it hard to mould and limiting its usefulness (Lataner, 1949).

More recently, Giddey and Dove (1991) incorparated water as a foam to develop the distribution of water. Foam preperation was applied by adding surfactants at level of 0.1-30% to water before whipping various gases such as air, carbon dioxide, nitrogen etc to make the foam homogenous. For viscosity enhancing 0.1-2% carbohdrates such as carrageenan, xanthan, pectin could be added to the foam as well. Chocolate produced thereof could remain solid up to 50 °C. However, this method needs some non-conventional equipments, leadings to high costs (Giddey and Dove, 1991).

In addition, since there are some more drawbacks in this method, such as removing desired flavor volatiles as well as high cost, time consuming procedures, risk of sugar bloom and poor quality final product; other researches have been proceeded to develop the HRC (Potter and Hotchkiss, 1998).

As regards with indirect incorporation of water technique, first study was implemented at 1921 and it succeeded that the chocolate can withdraw up to 49 °C.

Corn syrup and sucrose were utilized in the ratio of one-third and two-thirds respectively as sweetener as well as the chocolate was not subjected to conching after refining. So, sugar molecules remained partially uncoated by the fat in chocolate. When temperature exceeded the melting point of fat, fat liquification developed the sugar network and heat tolerance was attained. Also, any moisture found in chocolate helped to the formation of this sugar network. However, the chocolate manufactured has a poor texture, high viscosity, sugar bloom risk and relatively bad flavor owing to the lack of conching process as well as sweetener processing needs special equipments that lead to high cost and consume time (Friedman, 1921).

In time, many researches were made actual and similar problems obtained inspite of different recipes. High cost sourced by equipments, enzymes or humidity control necessity during whole processing as well as long time requirements, water absorption or sugar bloom due to polyols were faced (Stortz and Marangoni, 2011). So, techniques were improved in time and gel particules production were started to be applied. The technique of dispersing gel particules in cold polyol before adding it into molten CB is developed. Emulsion is formed by heating it above to dissolution temperature of gel particules that varied in the range 90-170 °C. This process generated the HRC up to 40 °C but it required 3-29 days to develop, depending on the formula. Also, gel that included water and polyol hardened within 3-5 days (Best et al., 2005).

As regards to addition of oil/fat binding polymers, a method consisting cornstarch or gelatin was implemented to produce HRC by Ogunwulu and Jayeola (2006). Melting point measurements were performed and increase was obtained by incorporation of starch or gelatin. It was also observed that the moisture content of corn starch chocolate increased, depending ong the increase in corn starch amount. In addition, chocolate made with 10% corn starch exhibited not significantly difference than conventional chocolate in terms of color, smoothness and taste. However, it reduced the sweetness of chocolate in sensory evaluation. On the other hand, chocolate made with 10% gelatin exhibited not significantly difference than conventional chocolate either in terms of color and sweetness. But, clearly poorer taste and decrease in smoothness were obtained in comparison of conventional one (Ogunwulu and Jayeola, 2006). Besides, accordance with researches addition of polymers such as starch, gelatin, milk proteins and β-glucans led to an increase in the chocolate viscosity and its heat resistance. Increase in viscosity was shown as responsible for maintaining chocolate's shape at high temperature (Stortz and Marangoni, 2011).

Recently, a novel method has revealed by Stortz and Marangoni (2011). According to their method, polymer ethylcellulose (EC) solution was used in production of chocolate and it was mixed with the chocolate after tempering until it becomed homogenous. Then, it was subjected to moulding, cooling and drying treatments and evoporating of the ethanol is provided. In the beginning, while there was a 10% solution of EC within ethanol mix, the final chocolate had 2% EC after drying. Evoporating ethanol treatment was actualized at 30 °C after 9 days storage process. After all, the final chocolate resisted against above 86 °C (Stortz and Marangoni, 2011).

Another study regarding addition of EC were revealed by Stortz et al., (2014). A solvent substitution method was developed to create HRC. Up to now, many studies have been produced about HRC, but the addition of EC in chocolate manufacturing is a novel technique. In conventional chocolate, it must be considered that the probability of an interaction among EC and a lecithin phospholipids is present at the sucrose surface. Since in the research, molecular interactions exhibited as an evidence of heat resistance for EC containing chocolate. The evidence proposed that, the sucrose found in chocolate had a major role in heat resistance. Sucrose crystals have an ability to interact with EC and generate an oil-trapping network which influences stability and provides strenght (Stortz et al., 2014).

One other well-known method to produce HRC is based on raising the fat melting point. Interesterification of fats or addition of a high-melting point fat are two approaches to achieve it (Stortz and Marangoni, 2011). Melting point of CB can be enhanced by direct interesterification by adding lipase enzyme. This method is fairly advantageous in the developing of HRC as well as sensory evaluation of interesterified product (Fennema et al., 2008). Previously, mahua and kokum fats were fractionated and blended (Jeyarani and Reddy, 1999). This method was shown to generate more HRC with good sensory features, but the heat tolerance of the chocolate was not ideal taking into account the melting temparature is only 34.8 °C with 5% kokum fat inclusion (Timms, 2003).

Last of all, some ingredients can be combined with CBS to improve the thermal stability of chocolate. In Peyronel and Marangoni (2014) study, the addition of sorbitan monostearate (SMS) to CBS was investigated. The base fat and different proportions of SMS were prepared and melting behavior, crystal structure and morphology were studied. Heat resistance at 40 °C with 25% SMS was evaluated at the end of the experiment. Authors had also stated that the absence of PGPR and lecithin may have significant implications in the final structure, since they seem to influence the lamellar structure which the SMS generated (Peyronel and Marangoni, 2014). In the research of Pernetti et al. (2007) studies, lecithin and sorbitan tristearate (STS) mixtures in sunflower oil was investigated. They showed that only STS was unable to gel the oils. But the addition of lecithin improved significantly the oil structuring ability of STS (Pernetti et al., 2007a; Pernetti et al., 2007b).

1.3 Workspace and Strategies

Initially, three strategies were defined in the project, targeting a new hard compound coating which stays stable in warm climate (35 °C) maintaining a good chocolate taste and mouthfeel. In accordance with outcomes of them, two more strategies were developed. Hereby, compound production was restricted to ball mill processing. An overview of the strategies is given below:

1. Adaptation of the fat composition (HPKOo or HPKS/HPKO instead of HPKS) enables to increase the heat tolerance of a dark compound coating up to 35 °C or higher.
2. The increase in heat tolerance by incorporating of a high-melting emulsifier such as mono- and diglycerides comprises the non-waxy mouthfeel.
3. The activation of hydrocolloids like MCC/CMC in the fat phase through high-shear mixing/ high-pressure homogeniser.
4. The objective is to increase the heat tolerance through the incorporation of polysaccharides, particularly starch, maltodextrine and hydrocolloids whether or not in combination with emulsifiers. To do list can be sorted as follows:

- ➢ Screening of starrier starches which are defined as milled, pre-gelatinised, native maize starchs that converts liquids and oils into free-flowing, non-sticky, dry powders to be used easily across a broad range of applications added before or after compound production or powdered between application and coating.

- Further screening of hydrocolloids which are alginates that are suspended in fat or in combination with water and sugar added to compound matrix, MCC/CMC/emulsifiers, MCC/CMC/modified starch and non-colloidal MCC vs. colloidal MCC.

5. The goal is to map if adaption of the processing (e.g. roll-refining vs. ball-refining, introduction of processing step to functionalise hydrocolloids, release of moisture from amorphous ingredients through directed heating prior application) can contribute to an increased heat tolerance of compound coatings. To achieve this, to do list includes comparison of heat tolerance of compound coating (HPKS, HPKOo and HPKS/HPKO) refined using rolls and balls exhibiting similar flow properties and fineness.

1.4 Project Hypothesis

The partial replacement of CBS (PKO, HPKS or HPKOo), the base fats of compound chocolate, by a higher melting fat in compound coatings will result in an increased heat tolerance, at least to some extent, without inducing a waxy mouthfeel.

Besides, the incorporation of a hydrophobic biopolymer will result in the creation of a secondary particle network supporting the structure of compound coatings, even though the fat phase is molten to a large extent.

Furhermore, alternative processing such as roll-refining followed by high-shear mixing allows to obtain compound coatings at reduced fat content and exhibiting similar flow properties. This will be suppose to improve the heat tolerance without affecting the sensory properties.

2. MATERIAL AND METHOD

2.1 Materials

In this research, PKO and its hydrogenated stearin and olein fractions (HPKS and HPKOo) were applied in a hard compound coating reference by (Belcolade Erembodegem, Belgium). The main CBS and its reference substitutes (R1 and R2) were obtained from local or abroad suppliers. Furthermore, some hydrocolloids and starches were added to the hard compound coating at a expense of sucrose at a 5% level.

2.1.1 Compound chocolate ingredients

Several ingredients utilising dark compound chocolate manufacturing are listed below.

- ❖ Different pH cocoa powders, i.e., 5.5 for natural cocoa, 7.8 or 8.3 for alkalised.
- ❖ Different types of emulsifiers, such as STS, PGPR, soy or sunflower lecithines.
- ❖ Colloidal or noncolloidal hydrocolloids, such as microcrystalline cellulose (MCC) and carboxymethyl cellulose (CMC).
- ❖ Several types of starches like starrier starch from Cargill, maltodextrine or dextrine.
- ❖ Sugar.
- ❖ Vanillin.

They were tested incorporated with oils both by alone or mixed. Table 2.1 shows the main ingredients which were tried in this project.

Table 2.1 : Variable ingredients of compound chocolates.

Fats	**Hydrocolloids**	**Emulsifiers**	**New Ingredients**
30% Fat	Fat based	STS	Starch
36% Fat	Water based	PGPR	Dextrine
		Lecithin	Maltodextrine

2.1.2 Laboratory equipments

During the analyses, several equipments were needed in terms of compound production. In the laboratory, a mixer, heating device, micrometer, ball mill refiner, microwave, thermometer, oven, shock cooler, timer, moulds for tablets, paraffin oil and DSC were used. Additionally, cake, muffin, biscuit, donut and wafer were utilised during application experiments.

2.2 Methods

2.2.1 Melting behavior and crytallisation measurement

In DSC analysis, the non-isothermal crystallisation behavior of CB and its blends in different proportions was recorded at using the Q1000 DSC (TA Instruments, New Castle, Delaware, USA), which is shown in Figure 2.1, equipped with a Refrigerated Cooling System. Nitrogen was utilised as purge gas. After temperature calibration, molten fat, approximately 10 mg, was sealed in hermetic aliminium pans and an empty pan was used as a reference (Tran et al., 2015). Then the pans were sealed by a pan crimper and samples were heated to 80 °C in the DSC instrument and kept for 10 min at this temperature. Cooling to –80 °C were actualized at a rate of 5 °C/min. At the end, the samples were heated to 80 °C at 5 °C/min, holding for 10 min and then cooled again to 20 °C at 5 °C/min. They were kept at this temperature for 120 min. Then, they were heated to 80 °C at 5 °C/min, kept for 10 min and then cooled again to 5 °C for 30 min at 5 °C/min. Finally, they were heated to 80 °C at 5 °C/min again. The thermograms of melting and cooling processes were recorded.

Figure 2.1 : Differential scanning calorimetry.

To determine the melting behavior and crystallization of vegetable fats, following steps were carried out.

- Several CBS that were first melted in the oven weighed in a precision scales and put into tubes.
- One drop from each oil tube was taken and dripped into hermetic pans. The pans were sealed and placed into DSC. Hermetic pans and sealing device are indicated in Figure 2.2 and 2.3.

Figure 2.2 : Hermetic pans and lids for DSC analysis.

Figure 2.3 : Hermetic pan crimper.

- DSC results were examined to see the thermal behavior.

2.2.2 Solid fat content measurement

The SFC profiles of vegetable fats were recorded using a 23 MHz 1 H pulsed nuclear magnetic resonance (pNMR) Maran Ultra device from Oxford Instruments (Tubney Woods, Abingdon, Oxfordshire, UK). Measurements were applied in triplicate. The oils were melted and filled into the tubes (10 mm outside dimension x 75 mm length x up to 3 cm height). Prior to measurement, the oils were firstly melted at 70 °C for 30 min, secondly chilled at 0 °C for 60 min and held at each measuring temperature for 30 min. The solids percentage was determined by pNMR. Data obtained may be identified as the ratio of the response consisting of all the hydrogen nucleic in the sample and response attained from the hydrogen nuclei in the solid phase. The direct display method was adopted during the measurements. In this method, magnetisation was sampled at two time periods – the first is shorter interval (11 μs) after excitation of the magnetisation and the second is longer interval (70 μs). The first measurement after the shorter period gives the magnetisation of both solid and liquid phase.

2.2.3 Compound chocolate production

To make compound chocolate, following steps must be carried out.

- Ingredients indicating on each recipe were mixed. If high shear technique will be used, oil and hydrocolloids were mixed for 10 min and added into other ingredients. Ingredient mix on the precision scales is shown in Figure 2.4.

Figure 2.4 : Ingredient mixing with precious scales.

- During mixing, a heater was worked to keep the mixture in the liquid state. Mixer with heater is shown in Figure 2.5.

Figure 2.5 : Mixer.

➢ After ingredient mixing, the chocolate was refined by ball mill refiner. To obtain high quality results, following steps must be performed into the ball mill.

- Ball mills must first be rinsed with specific oil which will be used in the current recipe for 2 min at speed 1.
- Ball mills must be cleaned with the test batch for 5 min at speed 5.
- Afterwards, ball mills which is shown in Figure 2.6 can be used to refine the “real” test batch for 15-20 min at speed 8.
- Once refining is done, the following out of the product can start.

Figure 2.6 : Ball mill refiner.

➢ At the beginning of the dropping process of the test batch, fineness must be measured with micrometer indicating in Figure 2.7 to determine if particle size of the compound chocolate is acceptable. Indeed, its smooth mouthfeel will depend on it. Particles higher than 30 μm can be detected by the human tongue and will induce a grainy, unpleasant texture to the product.

Figure 2.7 : Micrometer.

➢ Procedure is described as follows:

- Mix 50% product with 50% paraffin oil homogeneously.
- Make sure micrometer is completely cleaned.
- Place 1-2 drops of the product on the measuring surface of the micrometer.

- Gently close it until the security screw starts clicking, continue turning till hearing 3 clicks.
- Result can be read on the screen and should be within this range: 18-23 μm.

➢ At the ending of dropping process, chocolate is put into boxex and stored at room temperature. Figure 2.8 shows the final compound chocolate samples that were filled in the boxes.

Figure 2.8 : Compound chocolates.

2.2.4 Tablet chocolate and application trials

To make tablet chocolate, following steps were performed:

➢ Melt the compound chocolate prepared by ball mill into microwave gradually upto 50-55 °C. Never exceed 60 °C not to be burnt.

➢ During melting gradually, always mix the product.

➢ Firstly arrange microwave showing in Figure 2.9, as 30 s and check the status of melting.

➢ Then arrange it as 15 s and repeat until fully melted.

Figure 2.9 : Microwave.

➢ Wait the chocolate until decreasing to 42 °C. During waiting, mix constantly and check the temperature via thermometer.

➢ Clean deeply the moulds with cotton.

- Pour the chocolate into the moulds. Do not forget to shake effectively during moulding. Figure 2.10 demonstrates a tablet chocolate moulding.

Figure 2.10 : Tablet chocolate moulding.

- Place the moulds into shock cooler at 4 °C.
- Keep them for 30 min.
- Demould the chocolates. Put into plastic bags and store at 18-20 °C. A tablet chocolate after demoulding is shown in Figure 2.11.

Figure 2.11 : After demoulding the tablet chocolate.

- Before heating test, leave them 3-4 days for stabilisation and crystallisation.
- Before analysis, put the tablets into heating cabinet for 4 hours at 35 °C.

Preparation of applications was done by subsequent stages:

- Follow same steps with tablet chocolate procedures until temperature decreases to 42 °C.
- Dip the applications (donut, cake, wafer) into melted chocolate.
- Put them quickly into the fridge at 6 °C and apply a shock cooling for 10 min.
- Let the applications crystallize at room temperature for a while.
- Place them into plastic bags and store at 18-20 °C.
- Before heating test, leave them 3-4 days for stabilisation and further crystallisation.
- Before analysis, put the applications into heating cabinet for 4 hours at 35 °C.

2.2.5 Shelf life analysis

For shelf life testing, prepared applications and tablets were placed into plastic bags and packed by packing machine. They were subjected to temperature fluctuations from 20 °C and 33 °C during one-week period.

Before heating test, tablets and applications are either heated for 4 hours or for 1 night at 35 °C and directly evaluated at room temperature within 30 s. In heating test, stickiness and hardness are evaluated. For waxiness; tasting at room temperature were performed.

2.2.6 Evaluation criteria

Grades were performed in accordance with Figure 2.12 and 2.13.

Figure 2.12 : General scores of general tablet chocolates.

Figure 2.13 : General scores of applications.

"-" represents liquid phase.

"0" represents soft and sticky situation.

"+" represents deformable position.

"++" represents breakable, small snap, slight fingerprint status.

2.3 Experiments

2.3.1 Thermal behavior measurements

Melting temperature of CBS1, CBS2 and CBS3 were attained by DSC. After each one was subjected to DSC analysis, position of the peaks was attained as an average and standard deviation of measurements. The value obtained from DSC melting curve depending on the peaks reported as the melting temperature.

Second thermal analysis was performed to see what will happen to heat tolerance of CBS after adding an emulsifier. STS was used as emulsifier in the project. SFC% at 25 °C and melting temperature profiles for mixtures containing different percentages of CBS with STS using a DSC were determined. Heat tolerances of CBS were explained.

2.3.2 Heat tolerant compound chocolate trials

2.3.2.1 Several fat mixture experiments

First of all, various CB alternatives were prepared in definite proportions and fourteen different chocolates were produced. Tablets and applications were evaluated at 33 °C and 35 °C in terms of hardness and stickiness by touching.

Codes were given based on production dates. Mix 1, Mix 2, Mix 3, Mix 4, Mix 5, Mix 6, Mix, 7, Mix 8, Fat 1 (F1) and Fat 2 (F2) represent commercial fat names and each one was mixed with R1 or R2 or R3 in different proportions. R1, R2 and R3 represent different CBS that were evaluated for heat resistance. Coded recipes were explained in Table 2.2.

In addition, prepared according to results of previous experiment recipes are shown in Table 2.3. 8th recipe was produced again incorporated with or without a starch and the fat content was kept as 36%. 12th sample was produced again incorporated with CB alternative at the same proportion. But, both 30% and 36% total fat proportions in 12th recipe were evaluated.

Table 2.2 : Several chocolate recipes of experiment 1**.**

No	Codes	Explanation	STS%	F/H/E
1	CC090216.1	Mix1 with R1	1	F+E
2	CC090216.2	Mix2 with R1	1	F+E
3	CC090216.3	Mix3 with R1	1	F+E
4	CC090216.4	Mix4 with R1	1	F+E
5	CC090216.5	Mix5 with R2	1	F+E
6	CC090216.6	Mix6 with R2	1	F+E
7	CC060216.4	R1	1	F+E
8	CC070216.1	R2	1	F+E
9	CC211015.3	R2+R3	1	F+E
10	CC100815.3	Mix7+R3	0	F+E
11	CC080216.1	R1+R4	1	F+E
12	CC211015.4	Mix8+R3	0	F+E
13	CC080216.2	Cover adapted	1	F+E
14	CC060216.3	Cover	0	F+E

Table 2.3 : Several chocolate recipes of experiment 2.

No	Codes	Explanation	F/H/E	Fat%	PGPR%	Starch%
7	CC290216.3	R1	F+E	36	0	0
11	CC290216.7	R1+F2 (50/50)	F+E	30	0	0
11	CC090316.2	R1+F2 (50/50)	F+E	30	1	0
11	CC290216.5	R1+F2 (50/50)	F+E	36	0	0
12	CC090316.3	R2+F1 (97/3)	F+E	30	0	0
12	CC211015.4	R2+F1 (97/3)	F+E	36	0	0
8	CC090316.4	R2	F+E	36	0	2.5
8	CC070216.1	R2	F+E	36	0	0

2.3.2.2 Shelf life experiments

According to the results of Table 2.3, three CBS were chosen as applicable and shelf life analysis was done for them. Table 2.4 and 2.5 show the details of shelf life experiment for chosen CBS. Products were exposed to temperature fluctuations among 20-33 °C for one-week.

Table 2.4: Shelf life experiment of chosen CBS.

No	Code	Name	STS%	F/H/E	Fat%
1	CC290316.1	CBS2	1	F+E	36
2	CC290316.2	CBS3	1	F+E	36
3	CC290316.3	CBS1	1	F+E	36

Table 2.5 : Pictures of packed tablets for shelf life experiment.

CBS2	CBS3	CBS1
		

After heat stability testing to shelf life experiment products, another shelf life experiment was determined to be applied. This time, period detected as two-week and temperature fluctuations was not carried out. Products was kept under room temperature and results are recorded.

2.3.2.3 Hydrocolloid experiments

Regarding hydrocolloid experiment; firstly, CBS1 with three different hydrocolloids were used in compound chocolate production via Ultraturax (high shear) method and they were evaluated at 33 °C. Difference of this method is hydrocolloids are mixed with vegetable fat first by high shear mixer. And then mixture is added into ingredients of compound chocolate production. High shear technique includes hydrocolloid mixing for 5 min in melted vegetable fat. Table 2.6 demonstrates the recipes with hydrocolloids experiments and Table 2.7 and 2.8 indicates the recipes that are chosen and analysed depend on good results of Table 2.6.

Table 2.6 : Several chocolate recipes of first hydrocolloid experiment.

No	Code	Name	F/H/E	Fat%	STS%	Information
1	CC090316.1	CBS1	F+E	36	1	
2	CC120416.1	CBS1 + Hydroc 1 (34%/5%)	F+H+E	36	1	Colloidal (CMC)
3	CC120416.2	CBS1 + Hydroc 2 (34%/5%)	F+H+E	36	1	Non-colloidal (MCC)
4	CC120416.3	CBS1 + Hydroc 3 (34%/5%)	F+H+E	36	1	Non-colloidal (MCC)

Table 2.7 : Several chocolate recipes of second & third hydrocolloid experiment.

No	Code	Name	F/H/E	Fat (%)	Method
1	CC210416.1	CBS2+Hydroc1	F+H+E	36	Whisk
2	CC120416.1	CBS1+Hydroc1	F+H+E	36	Ultraturax
3	CC290316.3	CBS1	F+E	36	Whisk
4	CC210416.2	CBS2+Hydroc4	F+H+E	36	Whisk
5	CC210416.3	CBS2+Hydroc3	F+H+E	36	Whisk
6	CC070216.1	CBS2	F+E	36	Whisk

Table 2.7 recipes double checked to see the heating test duration. 4-hour heating stability test was performed in second experiment; 1-night heating stability test was performed in third one. Table 2.8 recipes analysed to prove the hydrocolloid success finally.

Table 2.8 : Several chocolate recipes of last hydrocolloid experiment.

No	Code	Name	STS%	F/H/E	Fluidity	Waxiness
1	CC290316.3	CBS1	1	F+E	OK	OK
2	CC290316.1	CBS2	1	F+E	OK	OK
3	CC290316.2	CBS3	1	F+E	OK	OK
4	CC210416.2	CBS2 + Hydroc 4	1	F+H+E	OK	OK
5	CC210416.3	CBS2 + Hydroc 3	1	F+H+E	Less fluid	OK

2.3.2.4 Starch layer experiment

Regarding layer coating of starch technique experiment; it is comprised within the framework of adaptation of processing studies. Difference of this method is starch is poured onto applications. Then the compound chocolate is poured onto applications that are shaked just after covered with chocolate.

Products are prepared like on the table below. 6th sample respresents new method. Difference of 5th than 4th is that, starch does not added while chocolate is being produced. 95% of chocolate is firstly mixed with 5% of starrier starch by mixer and applications are dipped into the chocolate later. Recipes of starch experiments are swown in Table 2.9.

Table 2.9 : Several chocolate recipes of layer coating of starch experiment.

No	Code	Name	STS%	F/H/E
1	CC290316.1	CBS2	1	F+E
2	CC290316.2	CBS3	1	F+E
3	CC290316.3	CBS1	1	F+E
4	CC290316.4	CBS2 with 5% starrier starch	1	F+E
5	CC040516.1	CBS2 + addition of 5% starch	1	F+E
6	CC040516.2	CBS2 + layer of starch	1	F+E

3. RESULTS AND DISCUSSION

3.1 Melting Profile and Solid Fat Content Results

Results of thermal behavior and SFC% measurements were shown in Figure 3.1, 3.2 and 3.3.

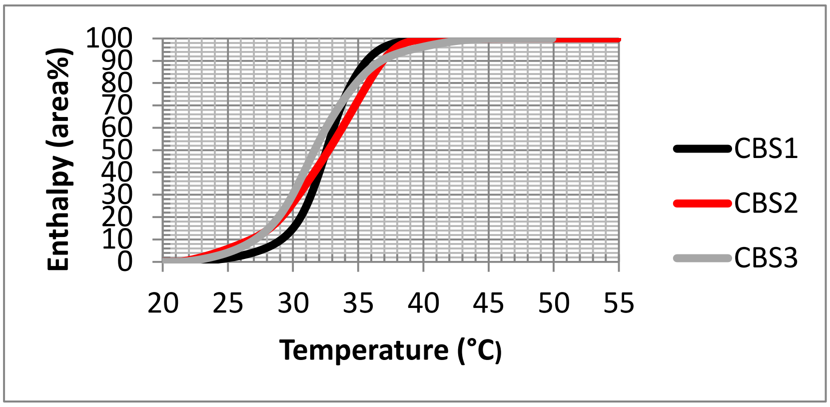


Figure 3.1 : Melting behavior of CBS after crystallised at 20 °C.

Figure 3.1 indicates that, chosen three CBS showed a high melting tendency after crystallised at 20 °C.

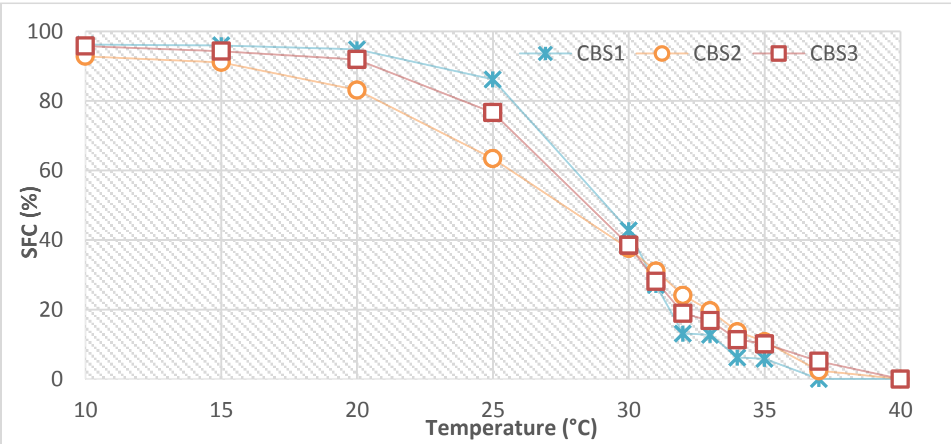


Figure 3.2 : SFC% of CBS.

Ideal case requires:

- Heat resistance: SFC as high as possible at required temperature 35 °C.
- No waxiness: SFC as low as possible at 37 °C.

➢ So, ideally huge drop in SFC from 35 °C.

Among 25-37 °C, cooling effect decreases with SFC%.

Among 31-35 °C, heat resistance increases with SFC%.

Among 35-40 °C, waxiness increases with SFC%.

A SFC of 40% or greater is observed in the temperature interval among 5 to 30 °C. The SFC is set to zero at the among 35 to 40 °C. According to diagram, CBS2 has the highest heat tolerance and low waxiness based on SFC%. CBS3 has relatively lower SFC% than CBS2. The diagram shows that, CBS do not resist against high temperature due to its low SFC value.

In accordance with second thermal analysis which are carried out on CB alternatives incorporating with an emulsifier, it was determined based on results that, the SFC decreased from 50% to 20% in the temperature interval among 30 °C to 35 °C, when the presence of STS is less 25%. Also, 100% STS kept its solid phase up to 45 °C. CBS containing among 5% to 25% STS gave a pleasant mouthfeel for humans. That showed that the usage of emulsifier increases the SFC without waxy taste.

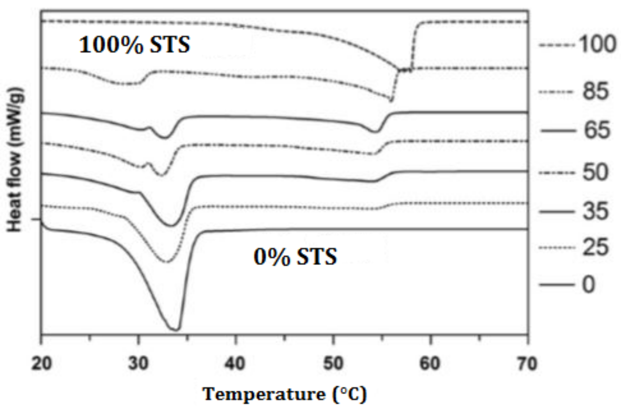


Figure 3.3 : Melting profiles for CBS with STS using DSC.

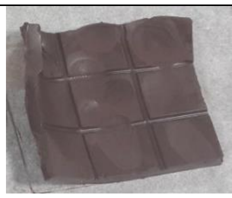
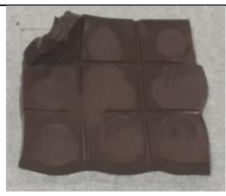
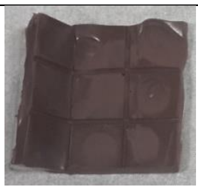
The melting curve for CBS demonstrated a broad peak melting temperature at 33 °C, while the melting curve for pure STS showed a peak at 55 °C. In addition, a third peak at 30 °C was remarkable when the concentration of STS was among 25% to 90%. As a results, DSC diagram proved the necessity of emulsifier to increase the melting temperature of chocolate. After screening and investigation of SFC diagrams, fat with required behavior does not exist. So, it is not possible to achieve 35 °C by playing only on fat.

3.2 Heat Tolerant Compound Chocolate Trials

3.2.1 Several fat mixture results

Regarding several fat mixture experiment, Table 3.1 shows the heat stability (HS) results of chocolates which are tried as first.

Table 3.1 : HS testing pictures applied to tablets at 33 °C and 35 °C.

No	**HS at 33 °C**	**HS at 35 °C**	**No**	**HS at 33 °C**	**HS at 35 °C**
1			8		
2			9		
3			10		
4			11		
5			12		
6			13		
7			14		

Scores of tablet chocolates were collected in Table 3.2.

Table 3.2 : Grades of tablet chocolates.

No	Codes	HS at 33 °C	HS at 35 °C
1	CC090216.1	(+)	0
2	CC090216.2	(+)	0
3	CC090216.3	(+)	(+)
4	CC090216.4	(+)	0
5	CC090216.5	(+)	0
6	CC090216.6	+(+)	0
7	CC060216.4	0	-
8	CC070216.1	(+)	(-)
9	CC211015.3	(+)	(-)
10	CC100815.3	+(+)	+(+)
11	CC080216.1	+	(+)
12	CC211015.4	+	(+)
13	CC080216.2	+	(+)
14	CC060216.3	-	-

We expect to get no fingerprint as much as possible on tablets. Also, hard and no sticky product at 35 °C was targeted. 3rd recipe is relatively better than 11th in terms of hardness and stickiness. But they both is not heat resistant at 35 °C.

10th recipe is the best among 14 recipes. However, since it is insufficient; we have to keep trying with new ones.

After tasting the tablets at room temperature, 13th recipe had a min waxyness and 14th recipe had no waxy mouthfeel.

Besides, results of heat tolerance of each application were shown in Table 3.3. Each scores were collected in Table 3.4 and average scores were collected in Table 3.5.

In accordance with tables, it can be summarised that, donuts were soft but not sticky. 14th recipe was the worst and sticky. 10th and 3rd products gave relatively good results for now.

As a result of first trial, applications gave better results than tablets, even at 35 °C. But, we have to create the best recipe for both.

Table 3.3 : HS testing pictures applied to applications at 35 °C.

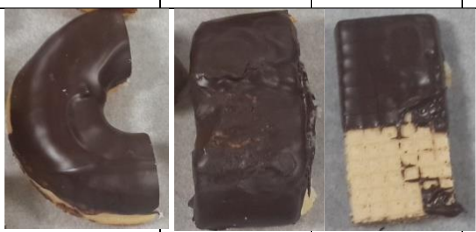
No	Donut	Cake	Wafer	No	Donut	Cake	Wafer
1				**8**			
2				**9**			
3				**10**			
4				**11**			
5				**12**			
6				**13**			
7				**14**			

Table 3.4 : Grades of applications.

	1	2	3	4	5	6	7	8	9	10	11	12	13	14
D[a]	++	++	++	++	++	++	++	++	++	++	++	++	++	-
C[b]	++	++	++	++	++	++	++	++	++	++	++	++	++	-
W[c]	+	+	+	0	+	0	+	-	+	+	-	+	(+)	-

[a]represents donut. [b]represents cake. [w]represents wafer.

Table 3.5 : Average grades of all applications.

No	Codes	HS at 35 °C
1	CC090216.1	++
2	CC090216.2	++
3	CC090216.3	++
4	CC090216.4	+(+)
5	CC090216.5	++
6	CC090216.6	+(+)
7	CC060216.4	+
8	CC070216.1	+(+)
9	CC211015.3	++
10	CC100815.3	++
11	CC080216.1	+(+)
12	CC211015.4	++
13	CC080216.2	+
14	CC060216.3	-

Scores of heat tolerance for second chocolate experiment were collected in Table 3.6.

According to results, tablets were worse and needed to be developed with aid ingredients. Applications were relatively better than tablets. 30% fat of 11th sample without PGPR and 30% fat of 12th sample gave little bit better results. However, we also have to consider easiness to apply and production cost. 30% fat content of both samples were quitely hard to apply. So we have to continue with 36% fat content. 8th sample with or without starch gave same result. To eliminate the F1 usage and provide cost reduction, we will proceed with 8th one instead of 12th.

Table 3.6 : Grades of tablets and all applications after second experiment.

No	Codes	HS for tablets at 35 °C	HS for applications at 35 °C	Easiness to apply
7	CC290216.3	-	-	0
11	CC290216.7	(+)	+(+)	-
11	CC090316.2	0	(+)	0
11	CC290216.5	0	(+)	0
12	CC090316.3	(+)	+	-
12	CC211015.4	0	+	0
8	CC090316.4	(-)	0	0
8	CC070216.1	(-)	0	0

Selected three CBS depending on Table 3.6 results were evaluated in Table 3.7.

Table 3.7 : General evaluation of chosen fats.

No	Codes	Fat%	Waxyness	Price	Comments
7	CBS1	36	No waxy	The most expensive	Taken as reference
8	CBS2	36	No waxy	-10% vs than No.7	
11	CBS3	36	No waxy	-5% vs than No.7	

3.2.2 Shelf life analysis results

Regarding results of shelf life experiment; compound chocolates were produced and tablets and applications were prepared and packed. They were subjected to temperature fluctuations from 20 °C to 33 °C and analysed after 1 week. Table 3.8 exhibits the results of one-week shelf life testing pictures.

After the products had been taken out from plastic bags, they were firsly evaluated before heating test. Table 3.9 shows the first evaluation of shelf life experiment.

According to results, donuts were in fairly poor position due to its greasiness structure itself. Wafers were in fairly good position. Neither oil nor chocolate were found on the pack. However, they cracked easily. CBS1 tablets had already stuck each other. Only CBS1 had deformation. CBS2 and CBS3 were acceptable status.

After tablets and applications had been exposed to 33 °C for 4 hour, they were evaluated for HS. HS pictures were indicated in Table 3.10 for chocolates that were exposed to shelf life analysis.

Table 3.8 : Pictures of tablets and applications after one-week shelf life testing.

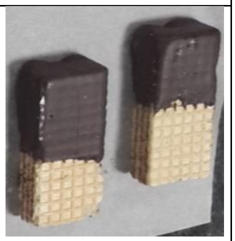
Name	Tablet	Donut	Muffin	Wafer
CBS2				
CBS3				
CBS1				

Table 3.9 : First evaluation of shelf life testing.

	CBS2	CBS3	CBS1
Muffin	+	(+)	0
Wafer	++	++	++
Donut	-	-	--
Tablets	+	++	-

Table 3.10 : Pictures of shelf life testing tablets after heated at 33 °C.

	CBS2	CBS3	CBS1
Tablets			

Scores of second evaluation of shelf life chocolates were collected in Table 3.11.

Table 3.11 : Grades of shelf life experiment after HS testing.

	CBS2	**CBS3**	**CBS1**
Muffin	+	+	-
Wafer	++	+(+)	(+)
Donut	--	--	--
Tablet	++	+	--

Depend on Table 3.11, except donuts, CBS2 gave the best results. two-week shelf life analysis was determined to be tested. Products did not subjected to temperature fluctuations during new shelf life testing. Muffins, tablets and wafers results were indicated in Table 3.12.

Table 3.12 : Pictures of tablets and applications after two-week shelf life testing.

Name	**Tablet**	**Muffin**	**Wafer**
CBS2			
CBS3			
CBS1			

Scores of two-week shelf life chocolates were collected in Table 3.13. According to results, they were in good positions at room temperature, they could be stabile when the conditions did not change. But the point was to keep them same after to be exposured to 35 °C. So, that was not possible in these circumstances.

Table 3.13 : Grades of two-week shelf life experiment.

	CBS2 at 20 °C	CBS3 at 20 °C	CBS1 at 20 °C
Muffin	+(+)	++	++
Wafer	+	+	+
Tablet	++	++	++
Tasting of tablets	No off taste	No off taste	No off taste

3.2.3 Cocoa butter substitutes general results

After all results so far, workspaces were reviewed and evaluated. For first strategy, results were summarised in Table 3.14.

Table 3.14 : Results of tablets and applications produced based on first strategy.

Code	Tablets at 33 °C	Tablets at 35 °C	Cakes at 33 °C	Cakes at 35 °C	Donuts at 33 °C	Donuts at 35 °C
CBS1						
CBS2						
CBS3						

Results were explained as follows:

- Better results only on cakes were obtained.
- There was oil interaction at the surface of the donuts.
- The most challenging applications were wafers.
- The worst results were tablets so it is not to focus anymore to tablets in strategy1.

In the beginning of the project, a lot of emulsifiers were tested but no good results were attained. In case of if coating was heat resistant at 35 °C, it was waxy as well. Both functionalities cannot be obtained at same time with emulsifiers. So, it was determined to proceed with other strategies to reach the target.

From the beginning until now, to do list comprises some ways to get convenient results which are indicated as follows. Also, new strategies were improved, targeting the project which is redefined to further increase the heat tolerance (≥ 35 °C).

3.2.4 Hydrocolloid analysis results

Hydrocolloids experiments was carried out to achieve the heat tolerance. HS results of hydrocolloid experiments were demonstrated in Table 3.15. Scores of first hydrocolloid experiments were collected in Table 3.16.

Table 3.15 : HS testing pictures applied to tablets and applications with hydrocolloids for first experiment at 33 °C.

No	Tablet	Donut	Cake	Biscuit
1				
2				
3				
4				

Table 3.16 : Grades of first hydrocolloid experiment.

No	Tablets at 33 °C	Biscuits at 33 °C	Donuts at 33 °C	Cakes at 33 °C	Average of all applications
1	0	0	0	0	0
2	(+)	(+)	+	+	+
3	0	0	0	0	0
4	(+)	(+)	(+)	(+)	(+)

As seen on the Table 3.16, second sample (CBS1+Hydroc1) gave only good results. Also, Hydroc2 changed nothing and Hydroc3 had a little effect. So, only Hydroc2 was excluded when preparing new recipes. New one focused to detect success of CBS2 with hydrocolloids. Each coded applications was prepared as follows before and after shock cooling. Figure 3.4, 3.5 and 3.6 show the tablets and applications during experiments.

Figure 3.4 : One of applications tray just before shock cooling.

After shock cooling, the products were holded for a while to crystallize.

Figure 3.5 : Pictures of applications before being packed.

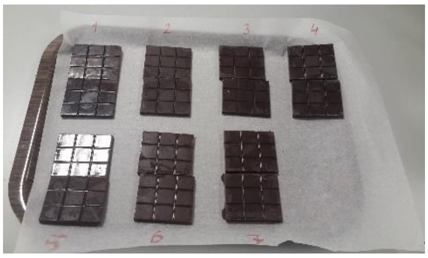


Figure 3.6 : Pictures of tablets before being packed.

Results of second hydrocolloid experiment at 33 °C were shown in Table 3.17 and scores were collected in Table 3.18.

Table 3.17 : HS testing pictures applied to tablets with hydrocolloids for second experiment at 33 °C.

No	Tablets	No	Tablets	No	Tablets
1		**2**		**3**	
4		**5**		**6**	

Table 3.18 : Grades of second hydrocolloid experiment at 33 °C.

No	Name	Cakes at 33 °C	Donuts at 33 °C	Wafers at 33 °C	Tablets at 33 °C
1	CBS2+Hydroc1	0	(+)	0	(+)
2	CBS1+Hydroc1	0	(+)	0	0
3	CBS1	0	0	0	0
4	CBS2+Hydroc4	+(+)	+(+)	+(+)	+(+)
5	CBS2+Hydroc3	++	++	++	++
6	CBS2	+	+	(+)	+

As seen on the result table at 33 °C, 5th recipe (CBS2+Hydroc3) gave the best result ever. Since, 4th, 5th and 6th recipes were visibly in good condition, they will be focused in heating test at 35 °C.

Results of same chocolates at 35 °C were shown in Table 3.19 and its scores were collected in Table 3.20.

Table 3.19 : HS testing pictures applied to tablets and applications with hydrocolloids for second experiment at 35 °C.

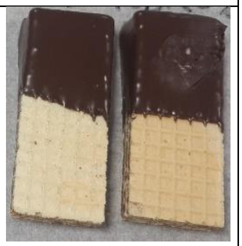
No	Tablet	Donut	Muffin	Wafer
4				
5				
6				

Table 3.20 : Grades of second hydrocolloid experiment at 35 °C.

No	Name	Cakes at 35 °C	Donuts at 35 °C	Wafers at 35 °C	Tablets at 35 °C
1	CBS2+Hydroc1	0	0	0	0
2	CBS1+Hydroc1	0	0	0	0
3	CBS1	-	-	-	-
4	CBS2+Hydroc4	(+)	+	0	(+)
5	CBS2+Hydroc3	+(+)	+(+)	+	+
6	CBS2	0	0	0	0

5th recipe (CBS2+Hydroc3) was in visibly good position, which is a proff that hydrocolloids must be utilised.

Heating test for 1 night at 35 °C was carried out to same six recipes above. Aim was to compare the grades with 4-hour heating test.

Scores of third hydrocolloid experiment was shown in Table 3.21.

Table 3.21 : Grades of third hydrocolloid experiment at 35 °C.

No	Name	Cakes at 35 °C	Donuts at 35 °C	Wafers at 35 °C	Tablets at 35 °C
1	CBS2+Hydroc1	0	0	0	0
2	CBS1+Hydroc1	0	0	0	0
3	CBS1	-	-	-	-
4	CBS2+Hydroc4	0	+	0	(+)
5	CBS2+Hydroc3	++	++	+	+
6	CBS2	0	0	0	0

As seen on the table one more time, 5th sample (CBS2+Hydroc3) was in visibly good position again. Duration of heating test did not change the result. We obtained a good outcome from compound chocolate with hydrocolloids.

After starch layer experiment which is expressed next section, making a last hydrocolloid analysis requirement had occured. Table 3.22 was demonstrated the results and 4-hour heating at 35 °C scores of final hydrocolloid experiment.

Table 3.22 : Grades of last hydrocolloid experiment at 35 °C.

No	Code	Tablet	Donut	Wafer	Cake
1	CBS1	-	-	-	-
2	CBS2	-	+	0	(+)
3	CBS3	0	0	0	0
4	CBS2 + Hydroc4	0	+	0	0
5	CBS2 + Hydroc3	+	+(+)	(+)	+(+)

5th recipe (CBS2+Hydroc3) gave the best result again among others. It also had non-waxy flavor. So, to get HRC for tropics, CBS2 with Hydroc3 is the best alternative for CB so far.

HS testing pictures implemented to both tablets and applications for last hydrocolloid experiment are collected in Table 3.23.

Table 3.23 : HS testing pictures applied to tablets and applications with hydrocolloids for last experiment at 35 °C.

No	Tablet	Donut	Muffin	Wafer
1				
2				
3				
4				
5				

3.2.5 Starch layer analysis results

Regarding starch layer experiments, results of layer coating of starch and testing pictures were demonstrated in Table 3.24, Figure 3.7 and 3.8 respectively.

Table 3.24 : HS testing pictures for starch coating at 33 °C.

No	Tablet	Donut	Muffin	Wafer
1				
2				
3				
4				
5				
6A				
6B				

Figure 3.7 : Addition of starch to chocolate for 5th recipe.

Figure 3.8 : Starch layer with applications for 6th recipe.

Layer coating of starch technique (6th recipe) was implemented to applications. Tablets were compared especially for addition of starch before/after chocolate production. Heating test for 4-hour at 33 °C were implemented to six different samples. Scores were shown in Table 3.25.

Table 3.25 : Grades of layer coating of starch experiment at 33 °C.

No	Code	Tablet	Donut	Wafer	Cake
1	CBS2	+(+)	+(+)	+	+(+)
2	CBS3	+(+)	+(+)	+	+(+)
3	CBS1	0	(+)	0	(+)
4	CBS2 with 5% starrier starch	+(+)	++	+(+)	++
5	CBS2 + addition of 5% starch	+(+)	++	+(+)	++
6A	CBS2 + thin layer of starch		+(+)	+	+(+)
6B	CBS2 + thick layer of starch		+(+)	+	+(+)

4th and 5th recipes were the best ones. So, different type of starch addition did not create any difference. New method was found worser than typical. So, heating test for 4 hours at 35 °C were evaluated to see the results of 4th and 5th recipes. Scores were shown in Table 3.26.

Table 3.26 : Grades of layer coating of starch experiment at 35 °C.

No	Code	Tablet	Donut	Wafer	Cake
1	CBS2	0	(+)	0	0
2	CBS3	0	(+)	0	0
3	CBS1	-	0	-	-
4	CBS2 with 5% starrier starch	0	(+)	0	0
5	CBS2 + addition of 5% starch	0	(+)	0	0
6A	CBS2 + thin layer of starch	0	(+)	0	0
6B	CBS2 + thick layer of starch	0	(+)	0	0

Aynone showed heat resistance. Hydrocolloids necessity was proven one more time.

3.3 Discussion

When examine the literature researches, even though HRC studies exist, any chocolate can produce which fully protects its structure against high temperature. It was obtained solid chocolates with some deformations at melting temperature of real chocolate, rather than fully melting. In this project, since the main target is to maintain the good chocolate taste and mouthfeel; stickiness, waxiness and hardness must be controlled and be same with the chocolate at room temperature after it was exposed to 35 °C.

Up until now, three main strategies were revealed about producing HRC. In present project, the strategies were improved, combined and implemented to compound chocolate. Enhancement of network microstructure was provided in the literature by second sugar network incorporating with water (Stortz and Marangoni, 2011). As well as high cost, this strategy has several drawbacks such as removing desired flavor volatiles, time consuming procedures, risk of sugar bloom and poor quality final product (Potter and Hotchkiss, 1998). So, this project comprised other two strategies' combination.

Addition of oil/fat binding polymer strategy has various achievements like exhibiting not significantly difference than conventional chocolate in terms of color, smoothness and taste. However, cornstarch or gelatin incorporation was inadequate in terms of reduce in sweetness of chocolate in sensory evaluation or clearly poorer taste and decrease in smoothness of chocolate respectively (Ogunwulu and Jayeola, 2006). Therefore, other hydrophobic biopolymers were investigated. CMC or MCC hydrocolloids, dextrines, starrier starchs were carried out to result in the creation of a secondary particle network supporting the structure of compound coatings.

Furthermore, according to litarature, oil/fat binding polymers led to an increase in the chocolate viscosity and its heat resistance (Stortz and Marangoni, 2011). However, at the same time, it must be considered that, hydrophilic sugar particules will lead to agglomeration and chocolate flow will be affected (Afoakwa et al., 2008a). So, hydrophobic emulsifiers such as lecithines, STS or PGPR were utilised in the project to provide arrangement in viscosity and heat tolerance since they enhance the chocolate texture and raise the fat crystallization rate (Garti and Aserin, 2012).

Third strategy to produce HRC is raising the fat melting point to attain heat tolerance. In the researches, interesterification of fats by adding lipase enzyme or addition of a high-melting point fat are two approaches to achieve it (Stortz and Marangoni, 2011). Although this method showed to produce more HRC with good sensory features, the heat tolerance was not ideal considering the melting temparature is around 35 °C (Timms, 2003). Since the project targets non-conching and non-tempering processes, lauric fats were preferred as CB alternatives which have higher melting point than CB.

The project reached to success taking into account all parameters and past inadequate studies. Hydrocolloid addition to CBS combination with emulsifier generated heat resistant compound chocolate which can resist against 35 °C and keep its shape, non-sticky structure, proper hardness, non-waxy taste and mouthfeel.

4. CONCLUSION AND RECOMMENDATION

Chocolate production is complex and needs various processes and technological operations to obtain the desired quality. During processing, rheological behavior, physical attributes and sensorial characteristics are impacted by its processing methods, ingredient composition and particle size distribution. Chocolate manufacturing comprises mixing, refining, conching and tempering processes if the chocolate is real and not to use for coating. Compound chocolate is chocolate-derived product consisting vegetable fats instead of cocoa butter. It is preferable alternative for real chocolate due to its low cost and being more applicable for coating purpose.

Chocolate consumers mostly expect from a chocolate to be had high quality assessment in features of flavor, aroma, mouthfeel and appearance. Also, warm climate consumers demand a more resistant chocolate against high temperatures. So, this project targeted to develop a new hard compound coaing which stays stable in warm climate (35 °C) maintaining a good chocolate taste and mouthfeel.

In accordance with this project, a heat tolerant chocolate can be made. This study showed the benefit of incorporating CBS for the development of heat resistant compound chocolate. In experiments, both tablets and applications were evaluated and it is exhibited that, CBS2+Hydroc3 was the most successful CB alternative for heat tolerance at 35 °C. It answered the flow requirements for enrobing and it is feasible without the necessity to temper.

Since the main point is to attain an inexpensive, non-waxy and heat resistant chocolate at the same time, further investigations should proceed even if a successful result obtained. Future studies should focus to attain much more feasible product for the industry. Also, products should be exposed to higher temperatures and investigations should concentrate to obtain resistance against 40 °C. Screening of maltodextrins exhibiting distinct properties in terms of impact on rheology and oil absorbing capacity should be researched and new workspaces should be revealed.

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