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# DETECTION OF RECOMBINED BUTTER BY DSC

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## Abstract

DSC can be used to quickly determine if a product labeled as butter is actually a recombined butter made without milk. Recombined butter is manufactured from anhydrous milk fat, skim milk powder, water, salt, and lecithin. Melting profiles of tempered samples of natural butter and recombined butter were alike, but DSC curves from 5 to 25°C of untempered refrigerated samples revealed that the enthalpy of the melting transition around 17–20°C was much higher for natural butter than for recombined butter. The procedure for differentiating the two products can be completed in less than 20 min.

**Keywords:** butter, butter oil, DSC, recombined butter

## Introduction

Recombined dairy products are manufactured in some developing countries where the fresh milk supply cannot keep up with the growing demand [1]. The main ingredients, skim milk powder, anhydrous milk fat or butter oil, and water, are combined and processed into the desired product. These ingredients can be stored at ambient temperatures, which is especially convenient in tropical and semitropical areas [2].

The manufacture of recombined butter is becoming an important option for places where local production of dairy products is desirable but refrigerated and frozen storage facilities are limited [3]. Standards for butter set by the Food and Agriculture Organization (FAO) of the United Nations apply to recombined butter as well [4]. Butter must contain at least 80.0% milk fat according to the FAO [4] and U.S. Federal and State laws [5]; the FAO also limits the water content to 16% and the nonfat milk solids content to 2% [4].

Cream is churned during manufacture of natural butter to break the oil-in-water emulsion and form a water-in-oil emulsion, which is necessary to prevent separation of the fat and aqueous phases during cooking [6]. In the manufacture of recombined butter, lecithin is added at a concentration of up to 1% to create the water-in-

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oil emulsion [3]. Lecithin, a phospholipid which is a component of all plant and animal membranes, is a powerful emulsifier containing hydrophilic and hydrophobic sites. In foods, lecithin adsorbs at the oil water interface, increasing the hydrophilicity of fat crystals and facilitating their suspension in water [7].

Rogers and Kieseker investigated the physical properties and flavor characteristics of recombined butter, adding lecithin to some samples to determine the effect on cooking properties [2]. Shukla *et al.* have recently related the rheological, compositional, and microstructural properties of recombined butters enriched with high melting triglycerides but made without lecithin [8–10]. They also obtained some melting profiles by DSC, which differed from those of typical recombined butter because of the high melting fat used [8].

On occasion, regulatory agencies must ascertain if a sample labeled as butter is actually a recombined butter. The procedure, which involves NMR analysis of an extracted sample to determine if lecithin is present, is expensive and time-consuming [11]. Tunick *et al.* developed a DSC method for examining imitation Mozzarella cheeses containing calcium caseinate and found that the emulsifying properties of the caseinate affected fat crystallization in untempered samples [12]. In this paper, we apply the same method to the differentiation of natural butter from recombined butter.

## Materials and methods

Butter oil and lecithin were combined by melting 200.0 g anhydrous butter oil (Land O' Lakes, Inc., Minneapolis, MN) on a hotplate at 40°C for 15 min, adding up to 2.45 g liquid soya lecithin (Modern Products, Inc., Milwaukee, WI) with stirring, and heating to 50°C. Then, 3.7 g skim milk powder (Darigold, Inc., Seattle, WA) and 5.0 g sodium chloride were dissolved in 39.0 g water with stirring. Recombined butter was prepared by pouring the two liquids into a stainless steel mixing bowl and beating with a KitchenAid electric mixer (Hobart Corp., Troy, OH) at medium speed for 5 min. The product was poured into a beaker and stored in a refrigerator at 4°C. The compositions of the recombined butters are shown in Table 1.

Three salted butter samples were purchased at local stores. Oil was extracted from one sample by melting the butter in a water bath at 39°C, centrifuging at 370×g for 15 min, and placing in ice to solidify the oil layer.

**Table 1** Composition and DSC data (mean±standard deviation) from untempered recombined butters

| Lecithin/ | Butter oil/ | Water/ | Salt/ | Solids[1]/ | Peak temp./ | Peak $\Delta H$/ |
|---|---|---|---|---|---|---|
| | | % | | | °C | J (g fat)$^{-1}$ |
| 0.03 | 80.7 | 15.7 | 2.0 | 1.5 | 19.5±0.1 | 0.11±0.01 |
| 0.12 | 80.6 | 15.7 | 2.0 | 1.5 | 19.6±0.2 | 0.15±0.04 |
| 0.49 | 80.3 | 15.7 | 2.0 | 1.5 | 19.4±0.2 | 0.16±0.06 |
| 1.00 | 80.0 | 15.6 | 2.0 | 1.5 | 19.5±0.2 | 0.18±0.02 |

Samples weighing 10–15 mg were placed in aluminum sample pans, immediately transferred to a Perkin-Elmer Model DSC7 differential scanning calorimeter (Perkin-Elmer Corp., Norwalk, CT) equipped with an Intercooler-2 subambient assembly, and scanned from 5 to 25°C at 5°C $min^{-1}$. These samples were considered to be untempered. Tempered samples were weighed into the pans and then dried in a forced-draft oven at 130°C for 20 min to eliminate interference from water on the DSC scans. The samples were tempered by holding at 50°C for 5 min to remove thermal history, cooling to –50°C at 5°C $min^{-1}$, and holding at that temperature for 15 min. Thermal profiles were then obtained by heating to 50°C at 5°C $min^{-1}$. Partial areas software, provided with the instrument, was used to determine the temperature and enthalpy ($\Delta H$) of selected features on the curves. The DSC was calibrated with an indium standard. Three replicates of each sample were analyzed.

## Results and discussion

Figure 1 shows the DSC curve of a typical tempered recombined butter sample. This characteristic melting profile of milk fat was nearly identical in all scans of natural butter, butter oil and recombined butter. The first valley in the curve occurred at 10.9–11.9°C, and the area of the curve to that point was 39.4–41.1% of the total area. The second valley was at 19.1–19.6°C and the area between the valleys was 21.0–22.7% of the total area. The thermal profiles were similar since the tempering procedure results in uniform crystallization of fat. These scans could not be used to distinguish natural butter from recombined butter.

Different results were obtained when the samples were scanned without tempering. This procedure allowed the thermal behavior of the product to be observed as it was warmed from refrigerator temperatures without permitting the fat to crystallize. The peak that had appeared between the two valleys was flattened when this

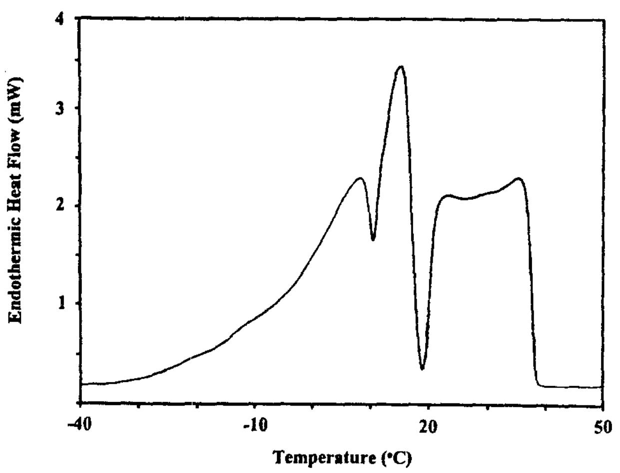


**Fig. 1** DSC melting curve of 13.1 mg of recombined butter containing 0.49% lecithin, after dehydration and tempering

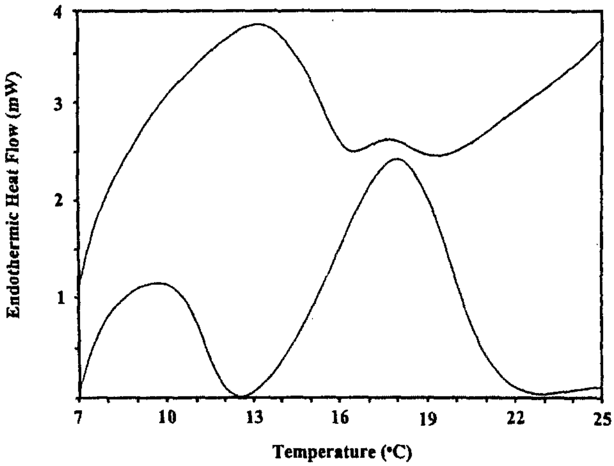


**Fig. 2** DSC melting curves of untempered samples of (top) 13.5 mg of recombined butter containing 0.49% lecithin and (bottom) 15.1 mg of butter

procedure was used (Fig. 2). The $\Delta H$ of this 17–20°C peak increased as the lecithin concentration, and presumably the level of emulsification, increased (Table 1). A comparison of Tables 1 and 2 shows that all of the $\Delta H$ values for the recombined butter peak around 17–20°C were much lower than the $\Delta H$ of natural butter. The anhydrous butter oil used to make the recombined butters had virtually no peak at 17–20°C (Table 2).

**Table 2** DSC data (mean±standard deviation) from untempere butter and butter oils

| Sample | Peak temp./°C | Peak $\Delta H$/ J (g fat)$^{-1}$ |
|---|---|---|
| Anhydrous butter oil | 19.8±0.1 | 0.033±0.003 |
| Butter extract | 18.9±0.3 | 2.16±0.16 |
| Butter (average of 3 retail samples) | 17.8±0.3 | 11.1±0.47 |

The state of dispersion of the fat phase in products containing milk fat affects melting properties [13]. Fat in butter consists of a crystal matrix of solid fat with some liquid fat [6]; between 6 and 30% is in the form of intact globules [13]. Anhydrous butter oil, which is at least 99.8% fat and contains no globules (6), would be expected to melt at a somewhat lower temperature when not tempered since it is more homogeneous. As a result, the portion of fat that melted in the 17–20°C range in natural butter melted several degrees earlier in recombined butter (Fig. 2). Laboratory extraction of the oil from melted butter yields a product that contains some fat globules and a small amount of water, and the $\Delta H$ of the peak from butter extract was between the values for natural butter and recombined butter (Table 2).

The results indicate that the nature of the fat in butter made from cream produced a sizable peak in the 17–20°C range in DSC scans of untempered samples.

This peak was easily distinguished from the small peak produced by recombined butters made from butter oil, milk powder, and water. DSC can quickly reveal the nature of a sample purported to be butter.

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