

Lipid polymorph impact on food quality: use to enhance Food Science education

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Chocolate – has a long shelf life...

or does it?

Polymorphism

Cocoa butter triglycerides (TAGs) have several ways to crystallize

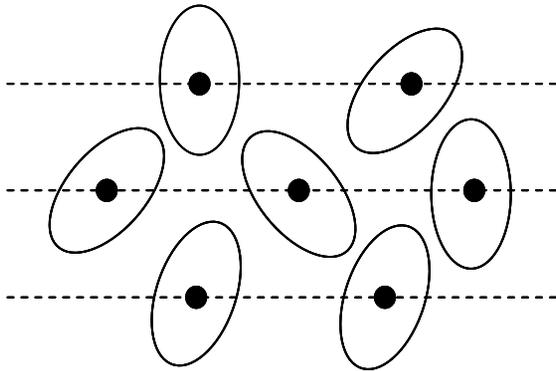
polymorphs

initially, lower stability polymorphs produced

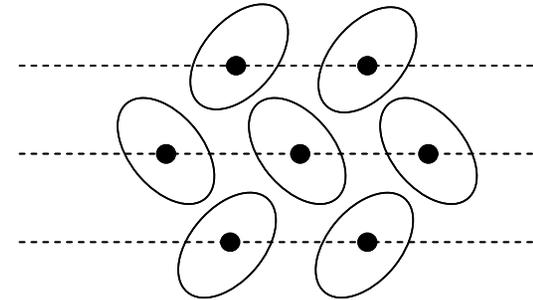
eventually transform to higher stability



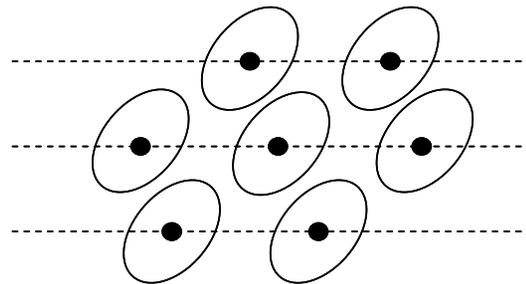
Cross sectional diagram of TAGs...



α polymorph (unstable)



β' polymorph (metastable)



β polymorph (stable)

Cocoa Butter Polymorphs

Cocoa Butter contains 40-50 triacylglycerols (TAGs)

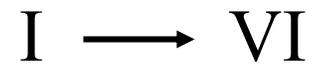
POP, POS, & SOS = ~80-90% TAGs

P = palmitate, O = oleate, S = stearate

$\alpha \longrightarrow \beta' \longrightarrow \beta$

rate of change influenced by amount of
liquid oil in sample

Polymorphs also expressed as I-VI



V/ β mp. 30-34°C

VI/ β mp. 32-36.3°C

Integrated proposal aims...

- Determine how storage affects quality of dark chocolate
texture, flavor and human perception
- Develop an understanding of role of lipid polymorph transition from V to VI on quality characteristics of stored dark chocolate
- Integrate an experiential learning experience into a summer workshop focused on using basic technologies to solve applied problems in FS

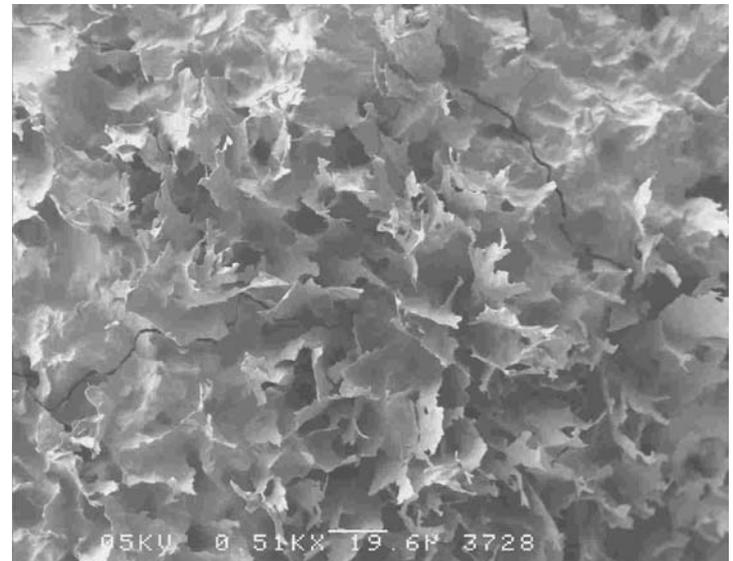
NAS – Announces need for K-12 education in sciences to enhance global competitiveness of US

Our study of the chemistry of chocolate during storage will be incorporated into experiential learning workshops for high school students and teachers

Fat Bloom

- Major cause of quality loss in chocolate industry
- Two theories:
 1. Polymorph transition from V to VI
 2. Increase molecular mobility – liquid migration to outer surface
 - Diffusion
 - Capillary flow

Figure 1.11 Scanning electron microscopy image of fat bloom.



Study of polymorphic transition and bloom characteristics

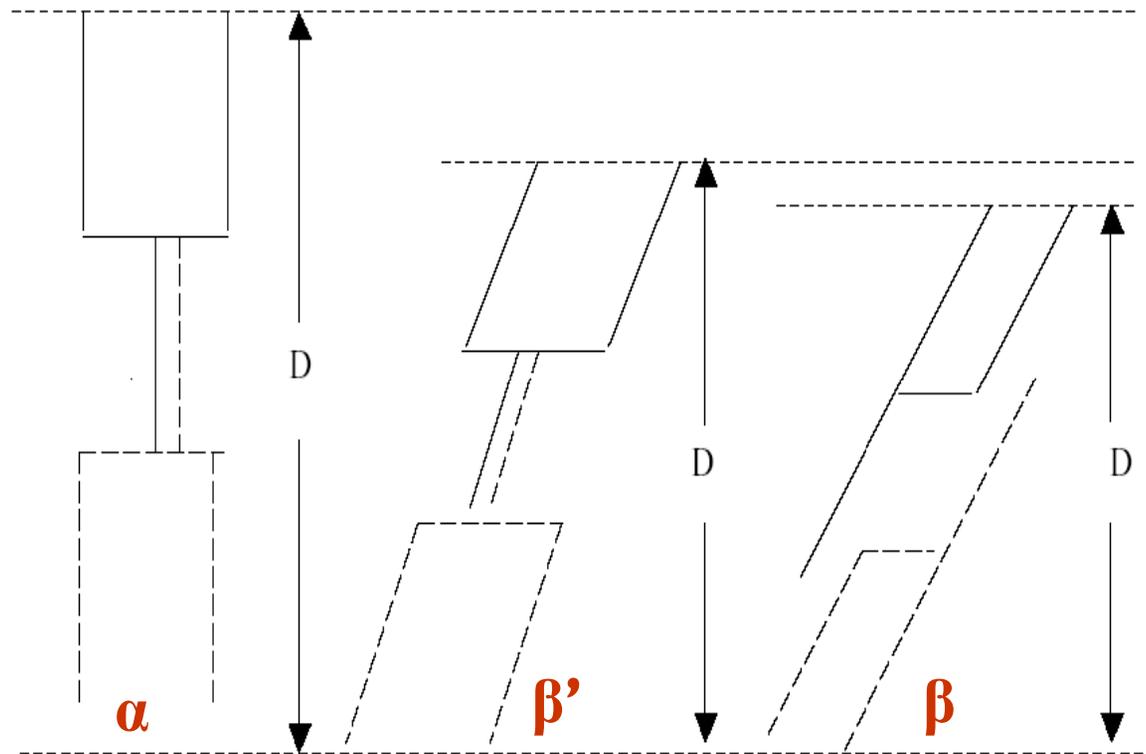
- Differential Scanning Calorimetry (DSC)
 - melting point characteristics
 - preliminary polymorph characterization
- X-ray Diffraction (XRD)
 - polymorph confirmation
- Atomic Force Microscopy (AFM)
 - topographical surface image
 - grain size/grain number

(Center for Microanalysis of Materials, UIUC)

Polymorphism

- Polymorphs identified by powder X-ray diffraction
 - d spacings

Triglyceride chain packing. ($D = d$ spacings)



Study of product quality (flavor and texture)

- sensory analysis (descriptive panel)
 - flavor and texture
- TA-XT2 texture analyzer
 - hardness, cohesive, adhesive, springy, gummy, and chewy
- dynamic headspace analysis/gas chromatography/mass spectroscopy (DHA/GC/MS)
 - flavor volatiles

Sensory analysis

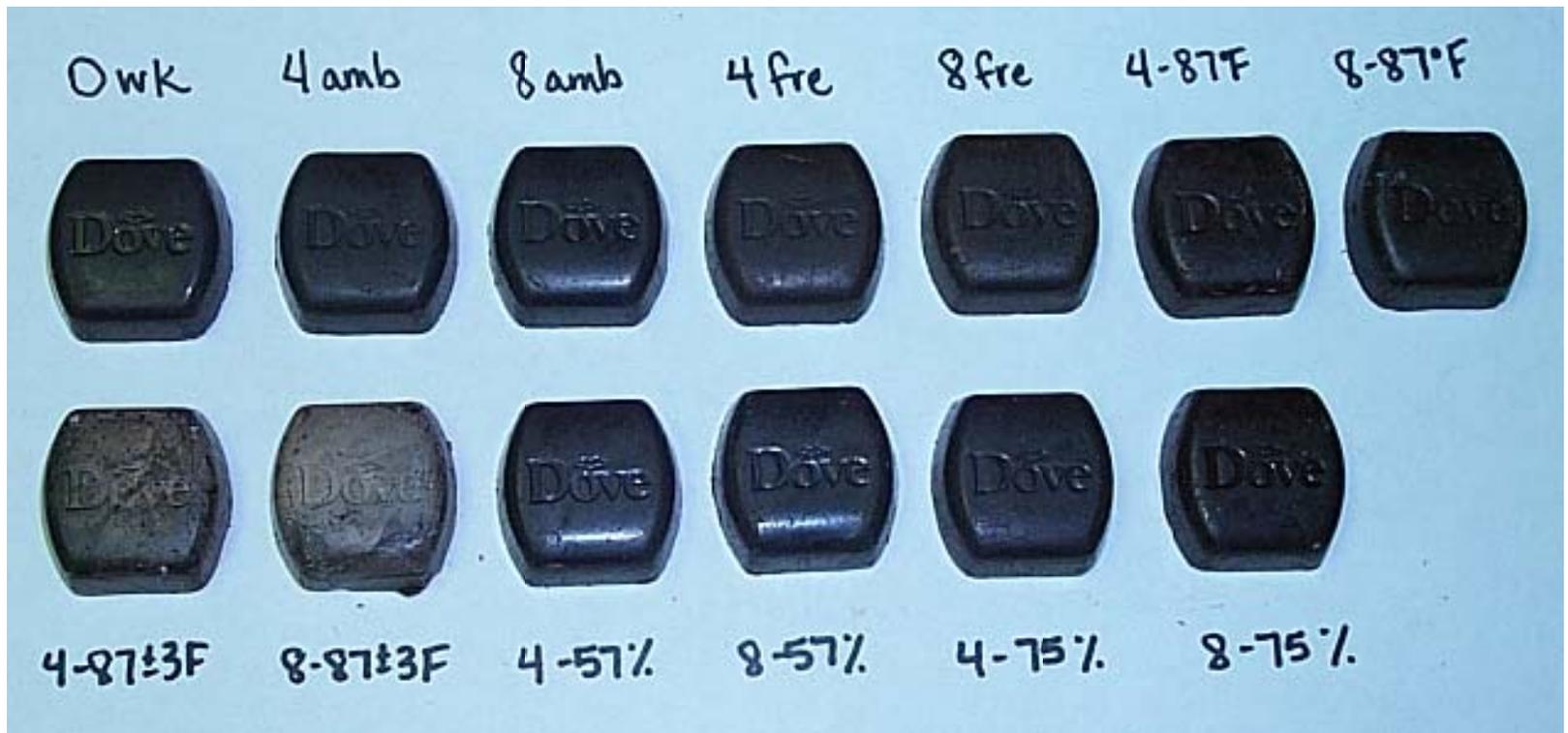
- Descriptive Sensory Panel
 - Twelve panelists
 - Three weeks of training for appearance and texture
 - One week of booth analysis
- Terms
 - texture: hardness, cohesiveness, chewiness, fatty mouthcoating, dry mouthfeel, toothpacking and melting
 - flavor: bitterness, sweetness, cream flavor, chocolate flavor, and roasted aftertaste

Chocolate storage conditions

| Condition | Temperature (°F/°C) | Relative Humidity (%) |
|--------------------------|--------------------------------|----------------------------------|
| Ambient (storage room) | 77.0/23.0 | 45.4 |
| Freezer | -17.0/-27.2 | 40.9 |
| Temperature fluctuations | 87.0±3/30.5±1.7 | 77.0 |
| High temperature | 87.0/30.5 | 44.1 |
| High relative humidity 1 | 77.0/23.0 | 57.6 |
| High relative humidity 2 | 77.0/23.0 | 75.3 |

Results

Effect of storage on appearance of chocolate.



Chocolate bar dimensions: 25mm long, 25mm wide, 10mm high

Instrumental textural analysis

- freezing – hardest, most cohesive, gummy and chewy
- high relative humidity – most adhesive

Polymorph/melting point analysis

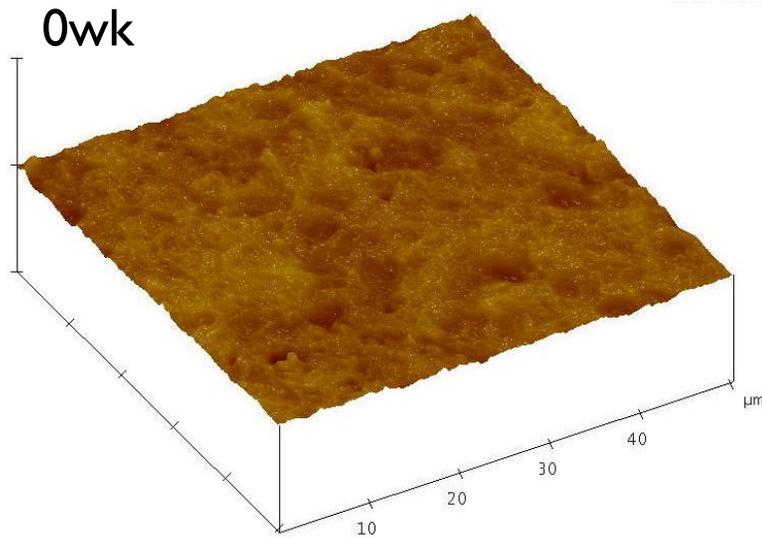
Polymorphism results by XRD and DSC (mean±SD)

| Sample | XRD Polymorph | Melting Point (°C) |
|----------------|---------------|------------------------------|
| 0wk | V | 33.5±0.5 ^{de} |
| 4amb | V | 34.0±0.0 ^{cd} |
| 8amb | V | 33.7±0.2 ^{de} |
| 4fre | V | 34.4±0.3 ^c |
| 8fre | V | 33.4±0.1 ^d |
| 4-87F | V | 35.4±0.0^b |
| 8-87F | VI | 35.8±0.2^{ab} |
| 4-87±3F | VI | 36.3±0.1^a |
| 8-87±3F | VI | 36.1±0.4^a |
| 4-57%RH | V | 33.7±0.2 ^{de} |
| 8-57%RH | V | 33.5±0.2 ^e |
| 4-75%RH | V | 33.5±0.2 ^{de} |
| 8-75%RH | V | 33.9±0.3 ^{cde} |

^{a-e}Numbers with same letters were not significantly different at $p \leq 0.05$.

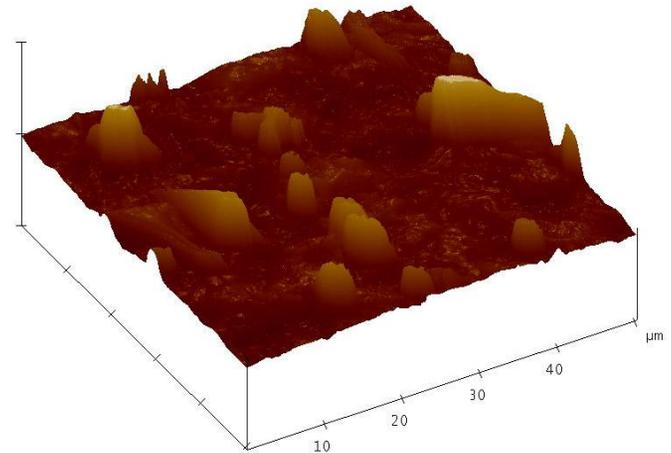
Atomic Force Microscopy (AFM)

Three dimensional AFM images of dark chocolate (z axis = 7 μm)

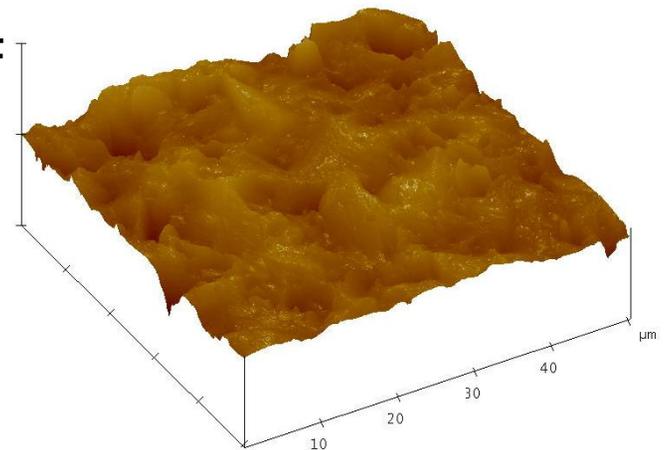


not stored

8-87F

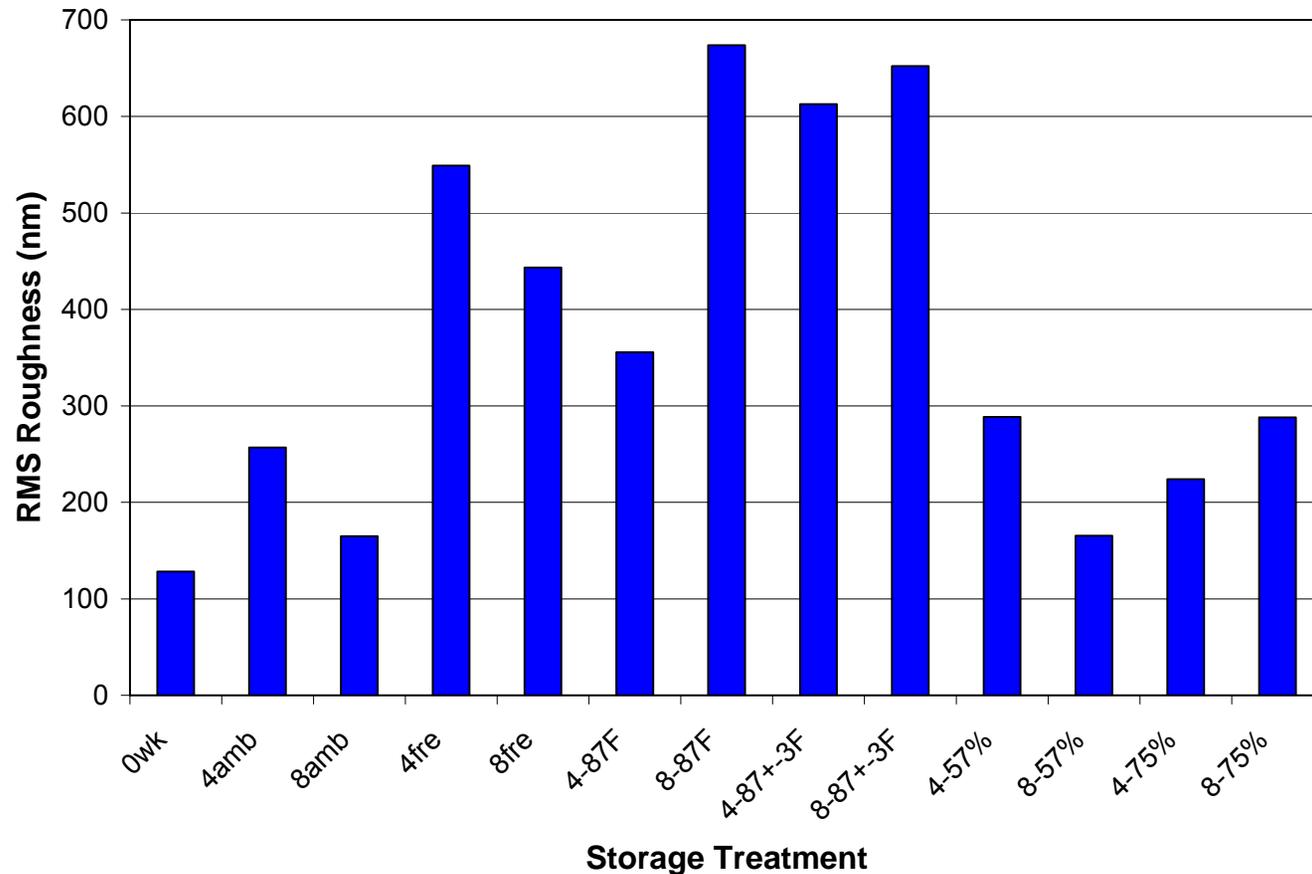


8-87 \pm 3F



Surface roughness – AFM

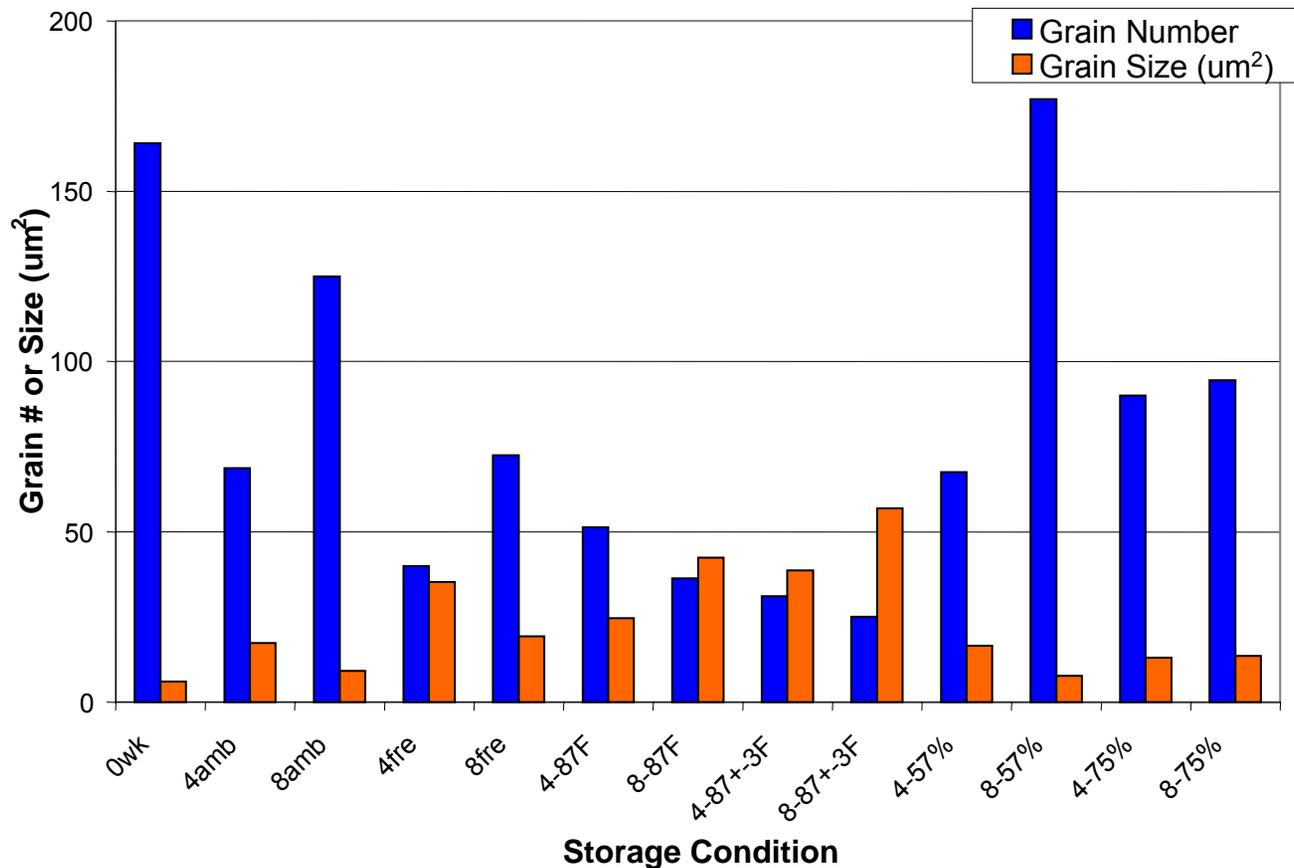
Atomic force microscopy measurement of surface roughness.^a



^aSurface roughness was significantly impacted by storage ($p \leq 0.05$).

Grain number and size

Atomic force microscopy measurement of average grain number and grain size.^a



^aGrain number and size were significantly impacted by storage ($p \leq 0.05$).

AFM related to human perception...

samples stored under conditions
(frozen and high temperature) leading to
transition to polymorph VI –

average grain number <37

average grain size >38 μm^2

**particles greater than 20 μm^2 detectable
by human tongue**

Sensory attributes of stored chocolate

8 of 12 attributes were significantly affected by storage

high temperature storage –
longer to melt, more toothpacking
(fluctuations – more toothpacking)

Sensory attributes of stored chocolate (cont'd)

chocolate not stored and stored
at ambient, frozen and high
relative humidity –

more cohesive, chewier, sweeter, with
higher dry mouthfeel and cream flavor
intensities...

How does storage impact the antioxidant capacity of chocolate?

Impact of storage on antioxidant capacity (ORAC) and procyanidin concentration

- Only samples stored at high relative humidity experienced reduction in antioxidant capacity.
- Procyanidin concentration was not significantly affected by any storage treatment.

Decrease in ORAC value without corresponding decrease in procyanidin concentration – implies other components (phenolics??) may be affected

Participation in Nano-CEMMS workshops
to encourage study of nanotechnology

Proposed educational workshops

- High school students
- Teachers

Goal –

attract young, bright high school students to the field of food science by the study of chocolate