SMALL-ANGLE X-RAY AND NEUTRON SCATTERING: APPLICATIONS TO FOOD

ELLIO T GILBERT
Leader, Food Structure and Dynamics
Leader, QUOKKA, Small-Angle Neutron Scattering
elliot.gilbert@ansto.gov.au
“The future development of food products will require an understanding of the relationship between nano-, supramolecular and higher order structures and of functionality on a physical, nutritional and physiological level……. food scientists and technologists will have to be engaged in nanoscience and nanotechnology” (Sanguansri & Agustin, Trends in Food Science and Technology 2006)

“Many of the structure-building elements in food are colloidal in nature and are built-up as a result of self-assembly of nano-sized molecules into particles or at interfaces. The next wave of food innovation will thus require shifting the focus from macroscopic properties to those on meso- and nano-scales as these subsequently control the hierarchical structures in food and food functionality.” (Ubbink and Mezzenga, Trends in Food Science and Technology 2006)
NUTRACEUTICALS & FUNCTIONAL FOODS

Food and Medicine……
RECENT EXAMPLES OF SANS (SAXS)

- Starch structure and gelatinisation
- Tannin nucleation and growth
- Casein micelle substructure
- Protein-polysaccharide complexes
- Encapsulation and controlled release
- Lipid metabolism and digestion
- Viscosity (bulk) to network structure/orientation (molecular)
A collaboration between the CSIRO and ANSTO, Australia and technology body, is trying to better understand the at the molecular level. It is hoped the research program scientists to design new foods to improve health and disease such as cancer and diabetes. Today the CSIRO agreement to use nuclear technology to first understand control the molecular architecture of food ingredients to nutrition. Scientists say ideally they want to be able to lower calorie version of any food. The research program foods that help in the fight against diseases such as cancer and diabetes. The OPAL facility online by the end of this year with food science research applications being conducted here. It is hoped the project the cutting edge of food science research.

Scientists to study food in bid to cure world ills

Australian scientists have embarked on a bold new project to solve some of the world's most pressing health problems by taking a closer look at food.

The country's top scientific and nuclear research bodies have joined forces to investigate the atomic structure of what we eat.

The cutting-edge collaboration between the Australian Nuclear Science and Technology Organisation and science body CSIRO, launched yesterday, aims to improve food safety, quality and nutrition.

But CSIRO food scientist Professor Peter Lillford said the project could have massive health implications if researchers figured out how to modify ingredients to lessen the risk of disease.

With ANSTO's new $300 million Opal nuclear reactor, researchers can use a technology called neutron scattering to look at materials 100,000 times smaller than the width of a human hair.

Professor Lillford said the plastics industry had long used the microscope to better understand the structure of the molecules they dealt with, but the food industry had been slow to follow suit. "The industry has been using hand lenses and low-powered microscopes for too long, but these are really no good for seeing inside what we eat," he said.

"Now we're being slowly dragged, kicking and screaming, into this area because we're too used to understanding the big, long, complicated molecules we're working with."

In particular, the researchers will look at the carbohydrate starch, and the role it plays in diabetes and cancer. Professor Lillford said the state of the starch molecule as it went into the stomach determined the amount of sugar in the bloodstream, and therefore the likelihood of developing diabetes.

It also determined the capacity for that starch to feed organisms in the bowel which protected against various cancers.

"There's obviously an urgent benefit here if we work out how to make food that still tastes nice but in fact lowers our sugar load in the bloodstream and also lowers the risk of cancer in human beings," he said.

Breads will offer cancer immunity

Matt Sun

Australian-made bread, cereals, biscuits or muffins could soon protect you from cancer.

Scientists will use a neutron scatterer at the Lucas Heights reactor to perfect a baking recipe that increases immunity to colorectal cancer.

The three-year project, announced today, aims to maximise the cancer-preventing abilities of starch.

Colorectal cancer kills one in 12 Australians every two hours and is the second-most deadly non-natural specific cancer after lung cancer. Australia has the third-highest rate of colorectal cancer, caused by diet deficiencies.

The $12 million project will be run by the Commonwealth Scientific and Industrial Research Organisation, the Australian Nuclear Science and Technology Organisation, and Food Science.

ANSTO scientist Dr Elliot Gilbert said the project would identify a component in starch that kills colorectal cancer.

There is a component we hope to identify in starch that, when digested, goes to the large intestine where it is broken down by gut bacteria. It then releases a component which kills colorectal cancer cells," Gilbert said.

The bread would also help reduce obesity and diabetes.

Nuclear recipe for cancer-cure foods

TORMANCE MENDEZ

Consumers will soon be able to choose a new range of cancer-fighting foods developed with Australian nuclear technology.

They aim to identify natural curers for cancer by bombarding slivers of food with neutrons from the nation's new 300 million Opal nuclear reactor in Lucas Heights, NSW.

Scientists want to discover the exact composition of some foods that demonstrate cancer-fighting qualities and make the recipe available to food manufacturers.

Project leader Elliot Gilbert, from the Australian Nuclear Science and Technology Organisation, says manufacturers could then include the ingredients in products.

"They will be able to say, 'if you eat this food you stand a chance of diverting colorectal cancer by X per cent,'" Dr Gilbert said.

ANSTO and preventative health researchers from CSIRO have teamed in the $10 million project that is expected to yield results in three years.

They will target resistance starch which is a component of starch that is not digested in the stomach or small intestine.

"In the large intestine it's broken down by bacteria and produces chemical components that actually kill colorectal cancer cells," Dr Gilbert said. "The food industry knows resistance starch does that but don't know what it is and don't know what the molecular structure is."

The study aims to unravel the long complex molecular structure of the ingredient by bombarding neutrons at slivers of food no thicker than 2mm and measuring how neutrons scatter to work out the structure of resistance starch.

He said the project aimed to give Australia a competitive advantage in food manufacturing. It was the first time nuclear science had joined the food industry.
OVERVIEW

• Some Quick Applications of SAXS/SANS
  – Proteins at low moisture
  – Organogelators

• SANS and Food Processing - RVA

• Starch and Enzymatic Hydrolysis
• Processing involves dehydration, subsequent re-hydration

• When proteins are dried for reasons of storage stability, the subsequently rehydrated forms do not exhibit same properties as original native states

• Kinetically-trapped structures from e.g. commercial drying

• Identify ageing (equilibration) mechanisms

• Relate molecular properties, microstructure to functional and bulk behaviour e.g. $T_g$ and molecular mobility

• Lead to formation of ‘Protein Syndicate’
  – Seven industrial partners $350K$ over 2 years

Energy transfer (meV)

- Red: vanadium
- Green, blue, cyan, magenta: 160K, 240K, 300K, 340K


25% H₂O, 320K
27% D₂O, 320K

27% D₂O
15.6% H₂O
21.5% H₂O
34.1% H₂O

temperatures - by DSC
MARGARINES AND ORGANOGELATORS

- Margarine is w/o emulsion (20-60% water)
- Fat crystal network provides firmness to product
- Melting behaviour determines mouthfeel mostly (solid/liquid)
- Is there an alternative to oil structuring by means of fat crystals?

![Chemical structures of γ-oryzanol and β-sitosterol](image)

*Courtesy – Arjen Bot*
STARCH

- Major storage polysaccharide of higher plants
- Second most abundant carbohydrate in nature next to cellulose
- 50-70% of dietary energy intake of humans
- Direct source of glucose, essential substrate in brain, red blood cells for generating metabolic energy
- Properties, interactions relevant to industry and human nutrition
Application of small-angle X-ray and neutron scattering techniques to the characterisation of starch structure: A review”, Jaroslav Blazek and Elliot Paul Gilbert*, *Carb. Polymers* 85 (2011) 281-293
RAPID VISCOANALYSER

• Tool for product development, quality/process control, QA.

• Cooking, stirring viscometer with ramped T and variable shear optimized for testing viscous properties of starch, grain, flour, foods.
NEUTRON PENETRATION

- if neutron were 1 cm across (pea)
- nucleus would be 10 cm across (ball)
- atom would be 1 km across

1 ms resolution of combustion engine
Brunner et al (TUM, PSI)
RAPID VISCOANALYSER

Design
Fabricate
Validate

Design
Modify
Shielding

[Image of the RIA 4500 instrument]
SIMULTANEOUS RVA AND SANS

Waxy Maize

\[ I(q) \]

\[ q (\text{Å}^{-1}) \]

Curve Fit Results

Wed, 21 Sep 2011 2:26:20 PM

Fit Type: least squares fit

Function: Sum_Model

Coefficient values ± one standard deviation

- \( K_0 = 0.0013117 \pm 0.000447 \)
- \( K_1 = 1.7638 \pm 0.0934 \)
- \( K_2 = 0 \pm 0 \)
- \( K_3 = 0.1533 \pm 0.00948 \)
- \( K_4 = 0.065659 \pm 0.000608 \)
- \( K_5 = 0.011126 \pm 0.0013 \)
- \( K_6 = 0.21792 \pm 0.00638 \)
H95 profile – 30 – 95 – 30 C over 13 mins

Waxy Maize
- Lamellar peak disappears after 4 min, correlates exactly with point in RVA at which the viscosity starts to increase markedly
- Subsequently, curve suggests formation of progressively larger scale fractal-like structures on 10 nm+ scale

Tapioca
Resistant starch

→ Fraction of starch that escapes digestion in the small intestine of healthy individuals

→ Bacterial fermentation in the colon into short chain fatty acids
Starch intake and colorectal cancer risk: an international comparison

A. Cassidy, S.A. Bingham & J.H. Cummings

Medical Research Council, Dunn Clinical Nutrition Centre, Hills Road, Cambridge CB2 2DH, UK.

Effects of resistant starch on the colon in healthy volunteers: possible implications for cancer prevention

Silke Hylla, Andrea Gostner, Gerda Dusel, Horst Anger, Hans-P Bartram, Stefan U Christl, Heinrich Kasper, and Wolfgang Scheppach

“The Diabetes Disaster that threatens to bankrupt Australia’s health system” – Sydney Morning Herald – 24th May 2008