Oxygen scavenging and aroma affecting enzymes embedded in barrier coatings
ENZYCOAT

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This presentation is addressed to Food Packaging and Paper Coating





○ Contain ○ Protect ○ Preserve

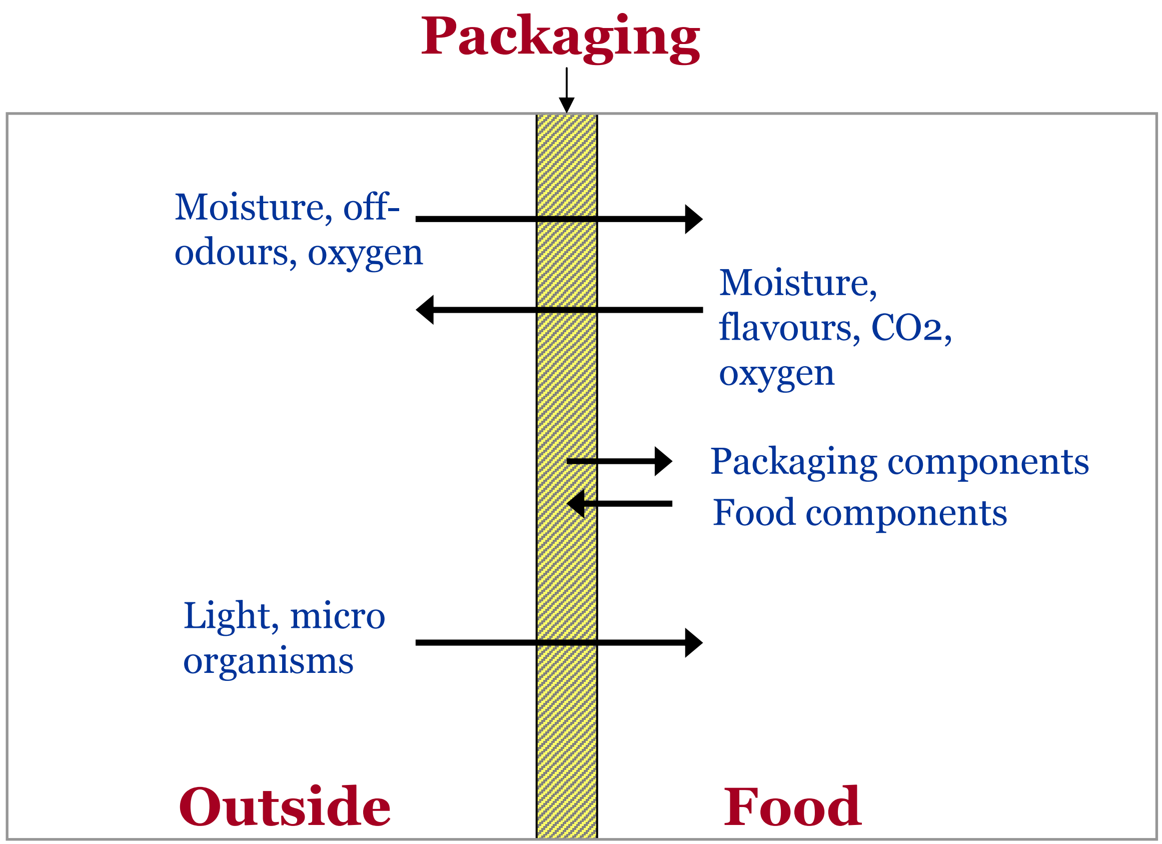
○ Inform ○ Communicate

○ Inexpensive ○ Environment






Food - Packaging Interactions



Absorption, migration, permeation

This presentation describes a method to produce *oxygen scavenging* paperboard, i.e. *active packaging*, through paper coating.

Why oxygen scavenging?

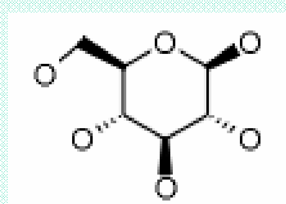
- Inhibit lipid oxidation
- Inhibit growth of aerobic microorganisms
- Prevent discoloration
- Prevent oxidation of vitamins

Method

- Scavenger - ferrous compounds
  - enzyme
  - ascorbic acid
  - oxidizable polymers
  - etc.

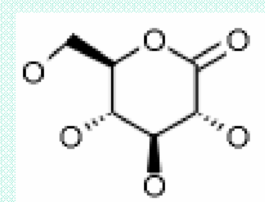
Oxygen-scavenging enzymes

Model enzyme: Glucose oxidase



β-D-glucose

+ O_2 —— ***glucose oxidase*** ——→



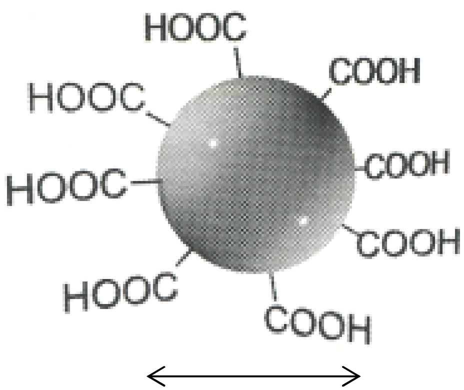
D-glucono-1,5-lactone

+ H_2O_2

H_2O_2 —— ***catalase*** ——→ H_2O + ½ O_2

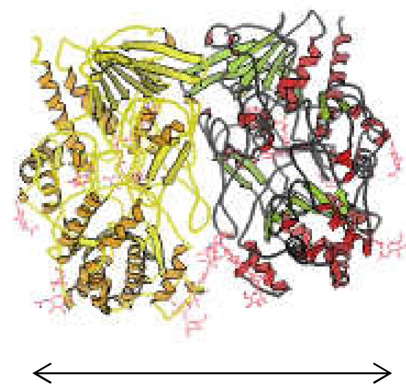
Oxygen Scavenging

Latex coating to which enzymes are added



Ca 200 nm

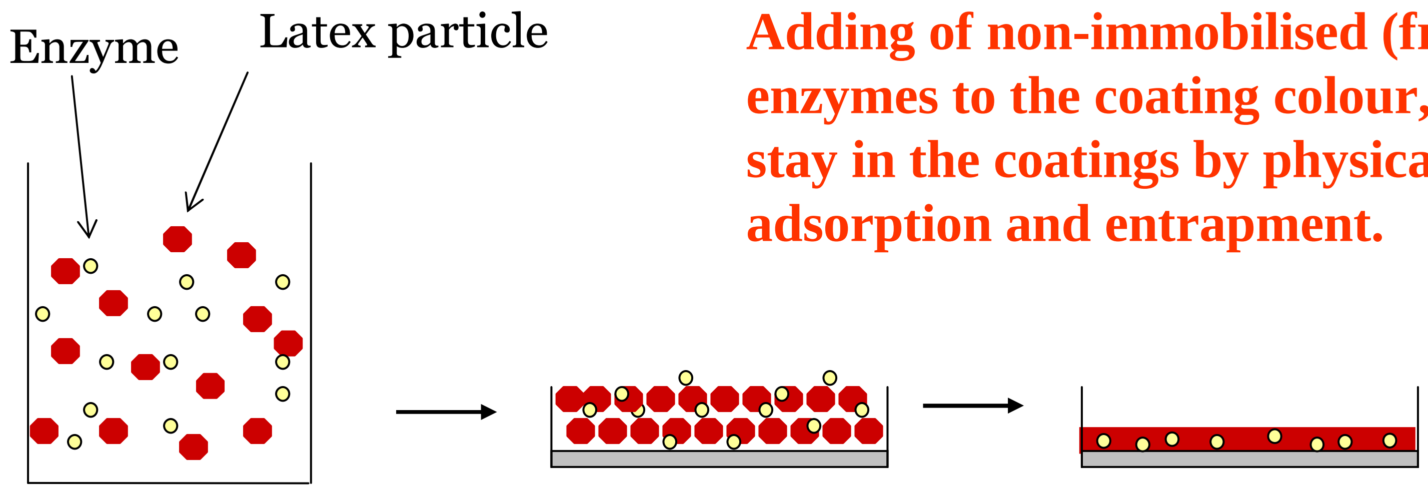
Styrene acrylate latex



8 nm

Glucose oxidase (GOx)

Entrapment of enzyme
(here exemplified by casting from solution)



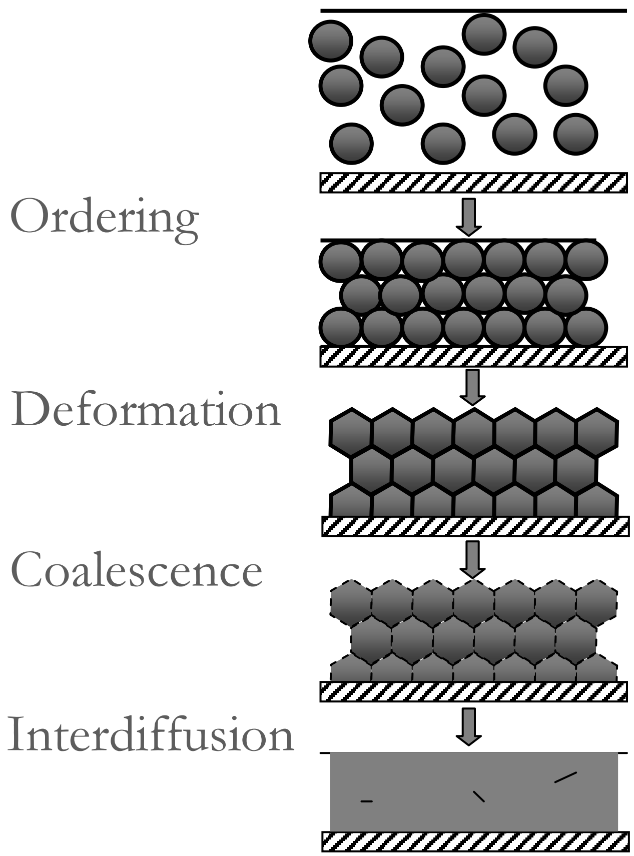
Adding of non-immobilised (free) enzymes to the coating colour, that stay in the coatings by physical adsorption and entrapment.

Enzymes added to the latex dispersion (coating colour).

Packaging of latex particles during drying.

Film formation during drying.

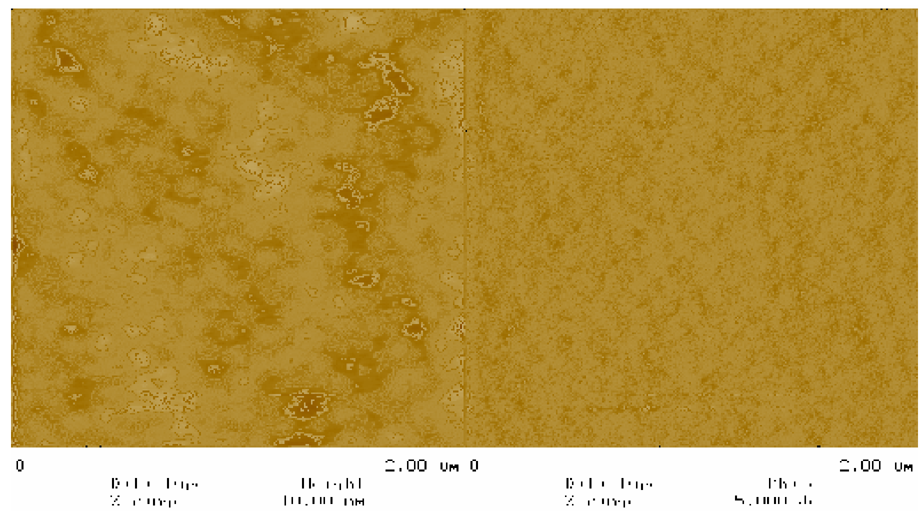
Film formation of latexes – a stepwise process



AFM imaging

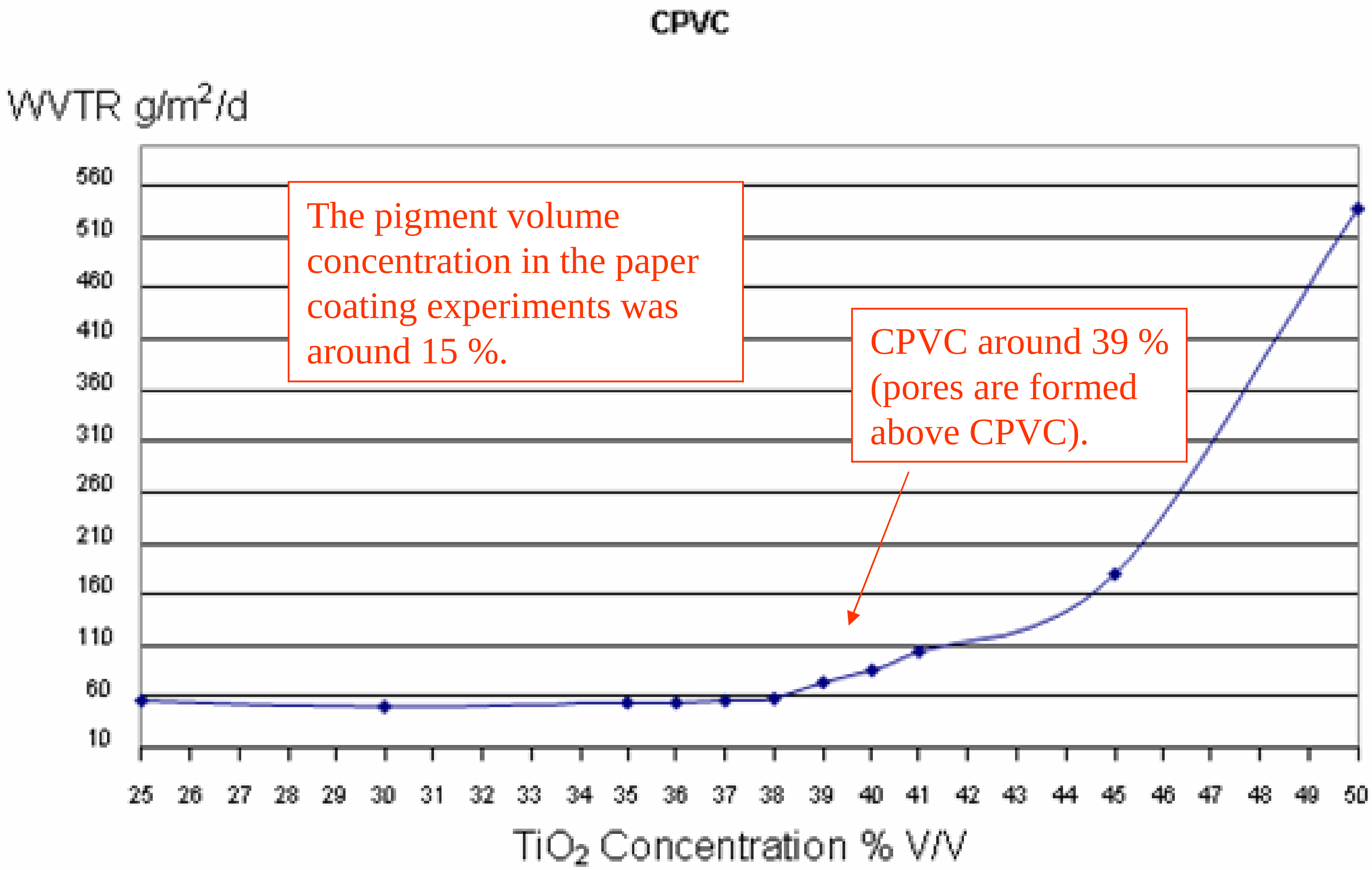
2 µm x 2 µm images recorded in Tapping Mode™

Topographic image **Phase image**

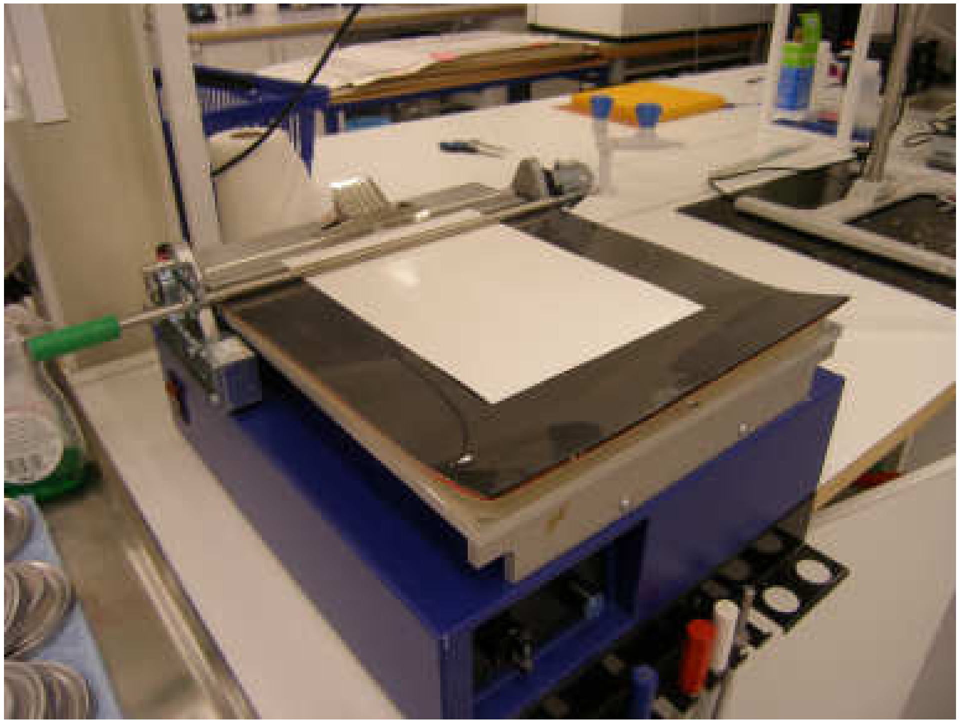


Latex films dried 20 °C above the glass transition temperature for 2 days. Still the shapes of the original latex particles is slightly visible.

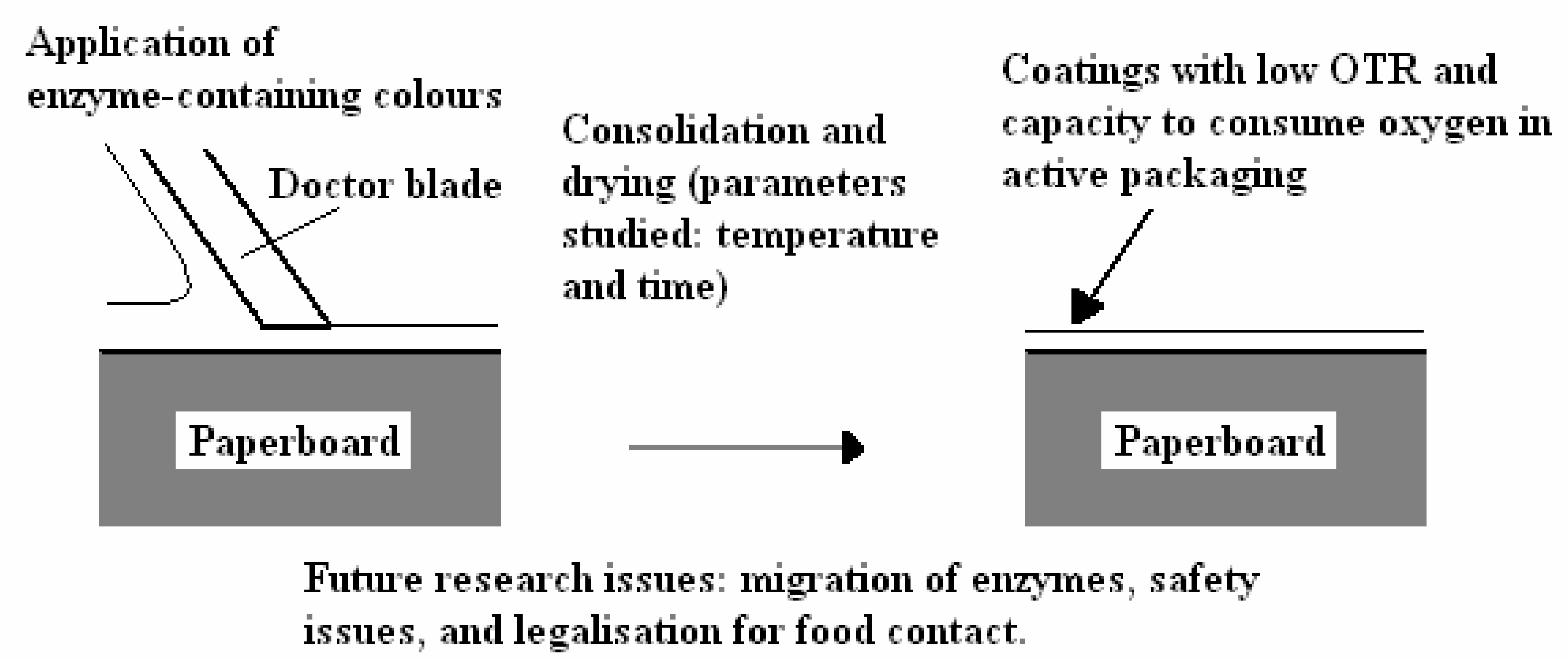
Porosity of coatings as a function of amount of pigment. Minerals can be used in order to prevent migration. Here TiO_2 (uncoated anastase, diameter 170 nm) was used.



The coating colours were draw down on paperboard by a laboratory-scaled bench coater.



Scope of the study



Results:
Coated paperboards

Paperboards used in the coating experiments

Enzycoat this side

Layer	Performa Natura (PN)	Natura Barr (NB)
Top coating	Double mineral coating	PE coating 20 g/m^2
Top layer	Bleached sulphate pulp	Bleached sulphate pulp
Middle layer	Bleached sulphate pulp + bleached CTMP	Bleached sulphate pulp + bleached CTMP
Back layer	Bleached sulphate pulp	Bleached sulphate pulp
Back coating	None	Multi-layer extrusion coating, PE/EVOH 56 g/m^2

Enzycoat this side

Coating colour formulations used in the coatings of paperboard

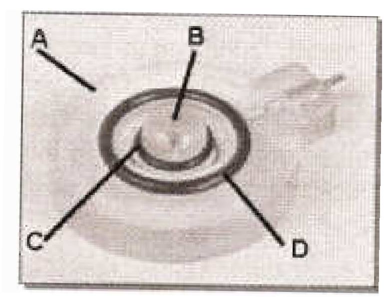
Enzycoat formulation

Component	**Amount in NaPA-containing colour pph**	**Amount in AMP-containing colour pph**
TiO_2	100	100
NaPA	0.36	0
AMP	0	0.36
Latex	134	144
GOx	1.4	1.5
Catalase	0.5	0.5
d-glucose	18	20

NaPA = Sodium polyacrylate; AMP = Amino-2-methyl-1-propanol

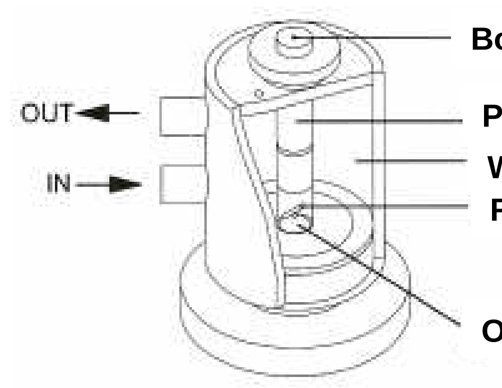
Measuring enzyme activity

The oxygen consumption was measured as reduction in oxygen activity in 3 ml of phosphate buffer (pH 7) containing 100 mM glucose when pieces of paperboard were immersed in the reaction vessel. Sample area: 1 cm x 2 cm.



Electrode disc

A **Dome of the disc**
B **Platinum cathode**
C **Silver anode**
D **Sealing**

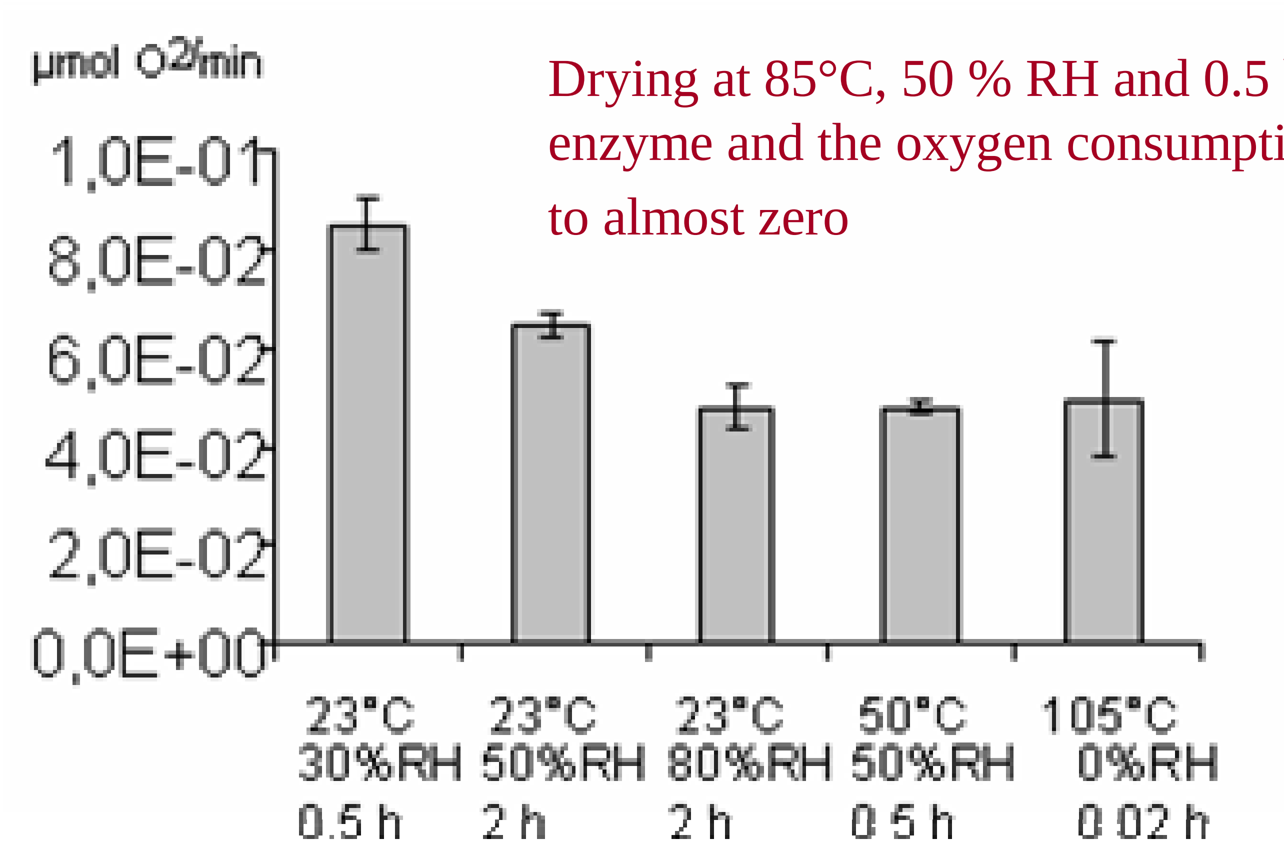


Electrode unit

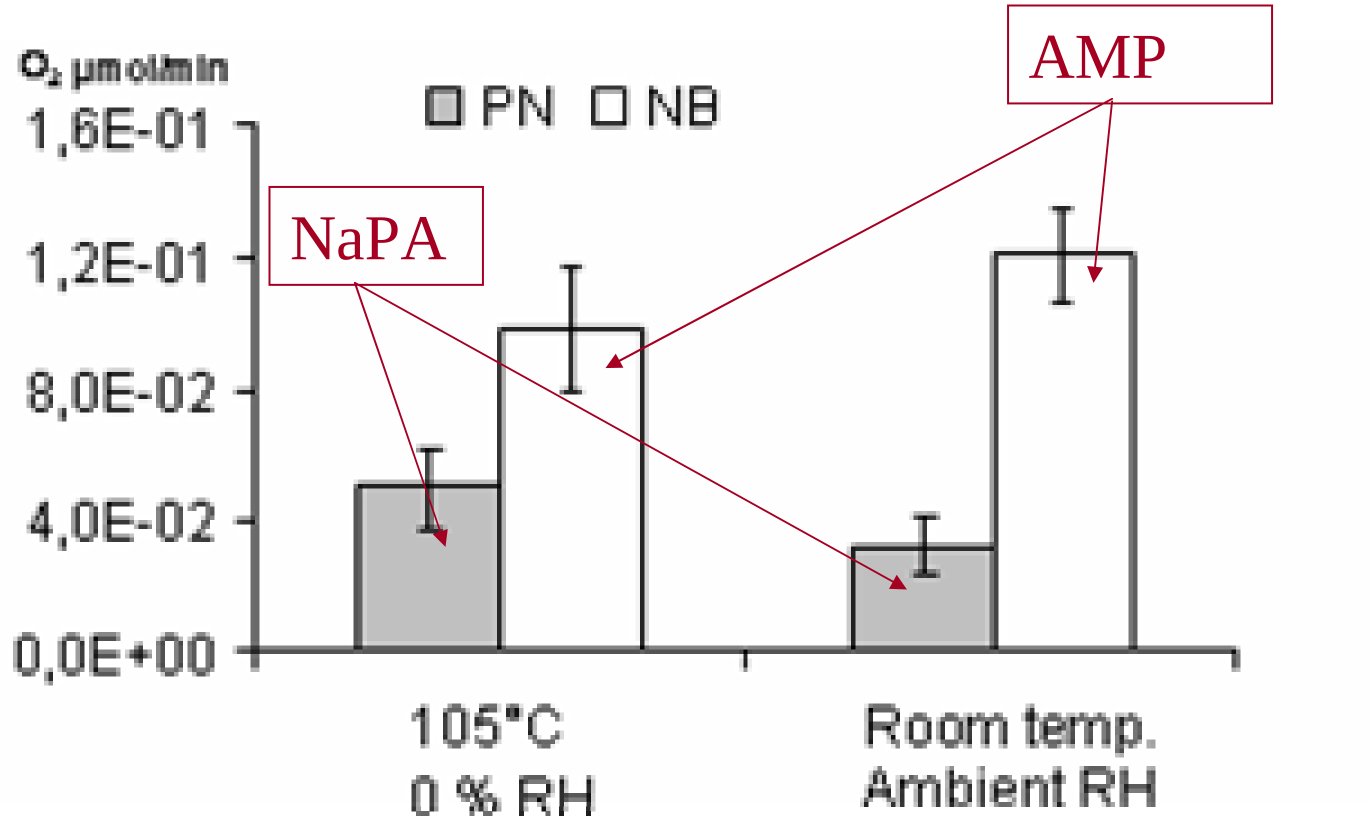
The current generated is proportional to the oxygen activity in the reaction mixture.

Enzyme activity at different **drying conditions**. Error bars indicate standard deviation of 3-4 samples. NaPA-containing colours coated on PN. Single coating and coat weights 29.7±2.9 g/m^2.

Drying at 85°C, 50 % RH and 0.5 h destroyed the enzyme and the oxygen consumption rate dropped to almost zero

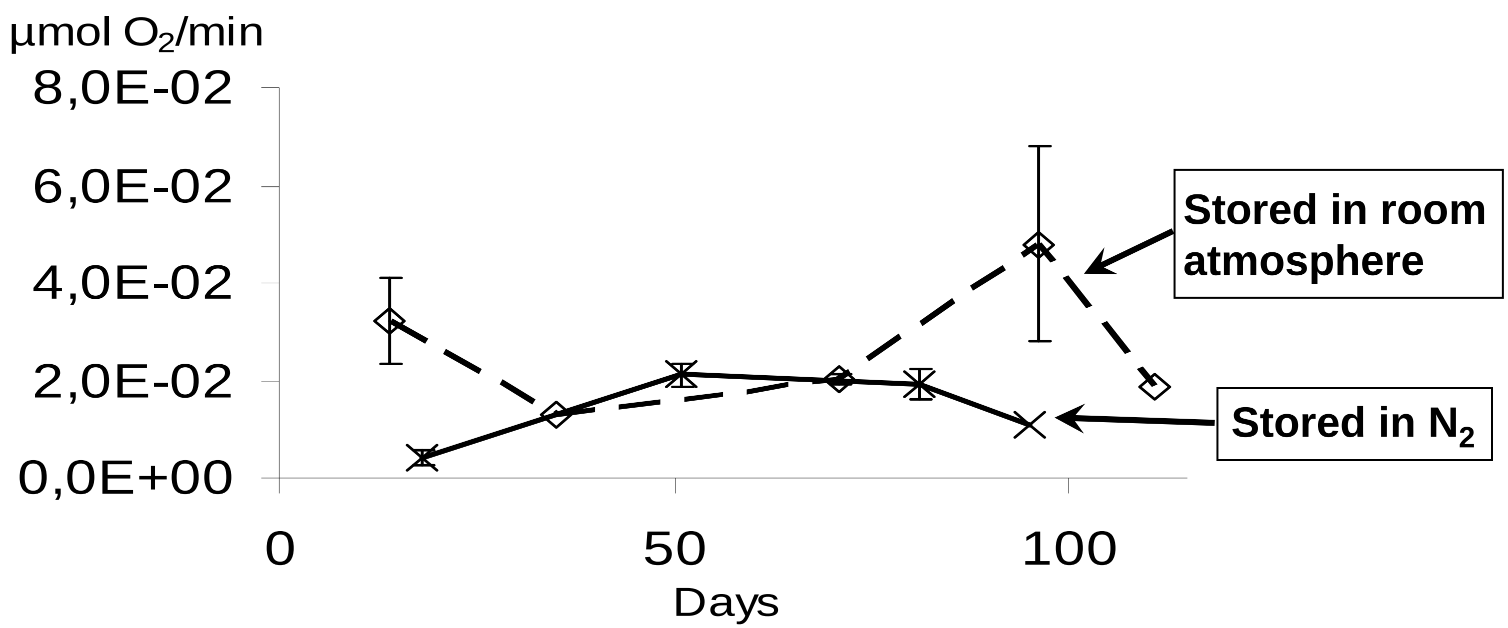


Enzyme activity at two different **drying conditions** and with two different **pre-treatments of TiO_2**. NaPA-containing coating on PN and AMP-containing coating on NB. Single coating and coat weights 27.7 ± 2.3 g/m².



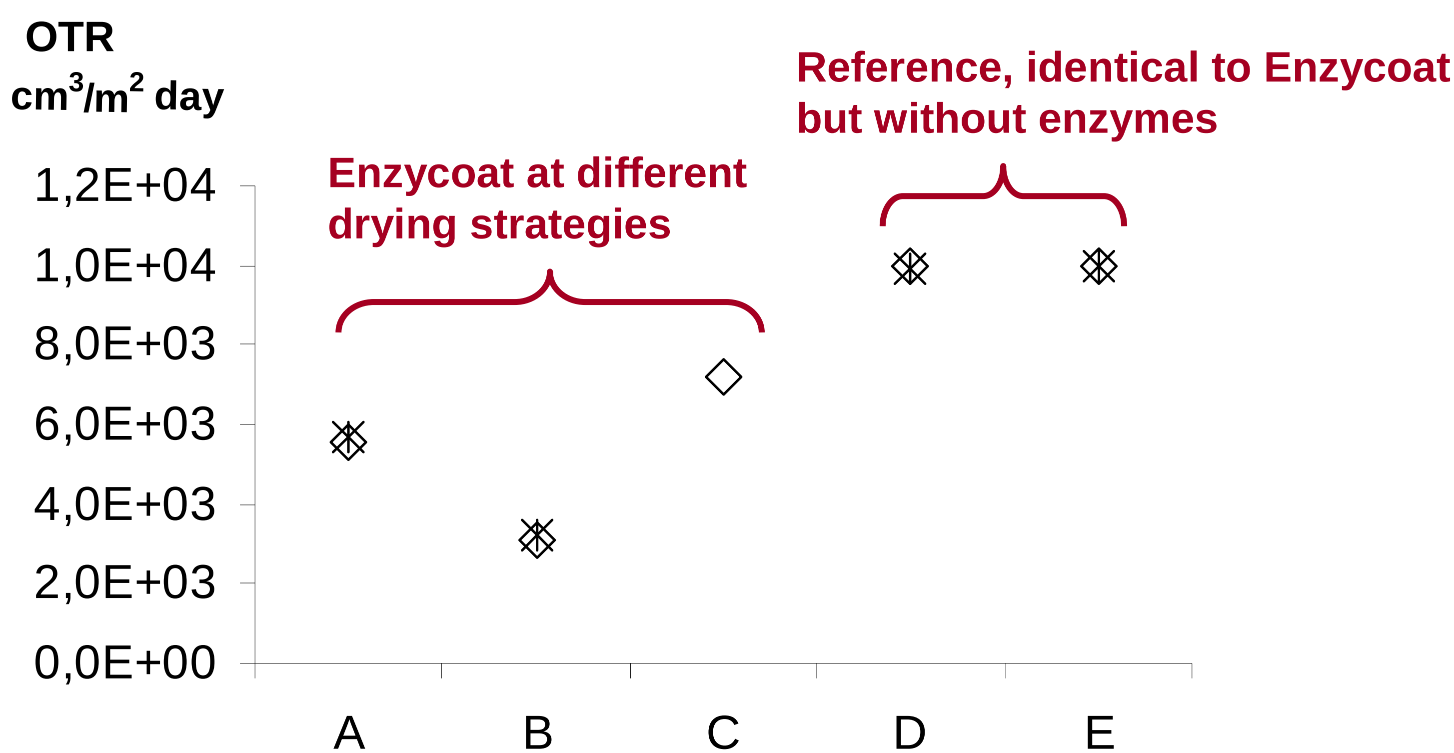
Effect of storage time

Oxygen consumption vs. days after application of NaPA-containing colours draw down on PN. Coatings dried at 21°C and N_2 atmosphere. Single coating and coat weight 24.1 ± 2 g/m^2.



Enzycoat layers on packaging board
Reduction of oxygen transmission rates (OTR)

coat weight 27 g/m² ± 3 g/m²

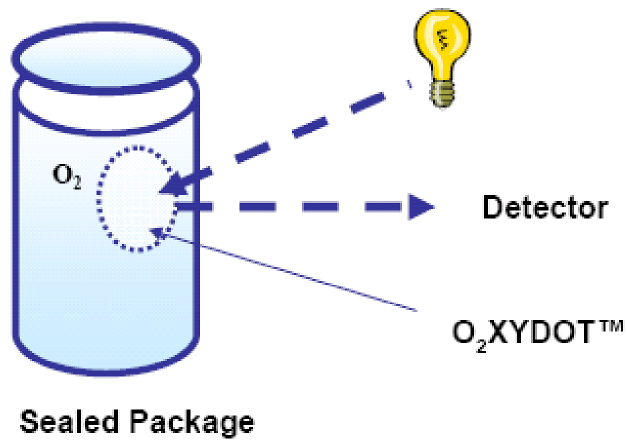


The packaging board coated with the enzyme-containing Enzycoat layer had better barrier properties than the corresponding enzyme-free product.

Results:
Oxygen absorption in sealed packages

Measuring oxygen reduction inside a sealed package

OxySense®
by DANISH TECHNOLOGICAL INSTITUTE



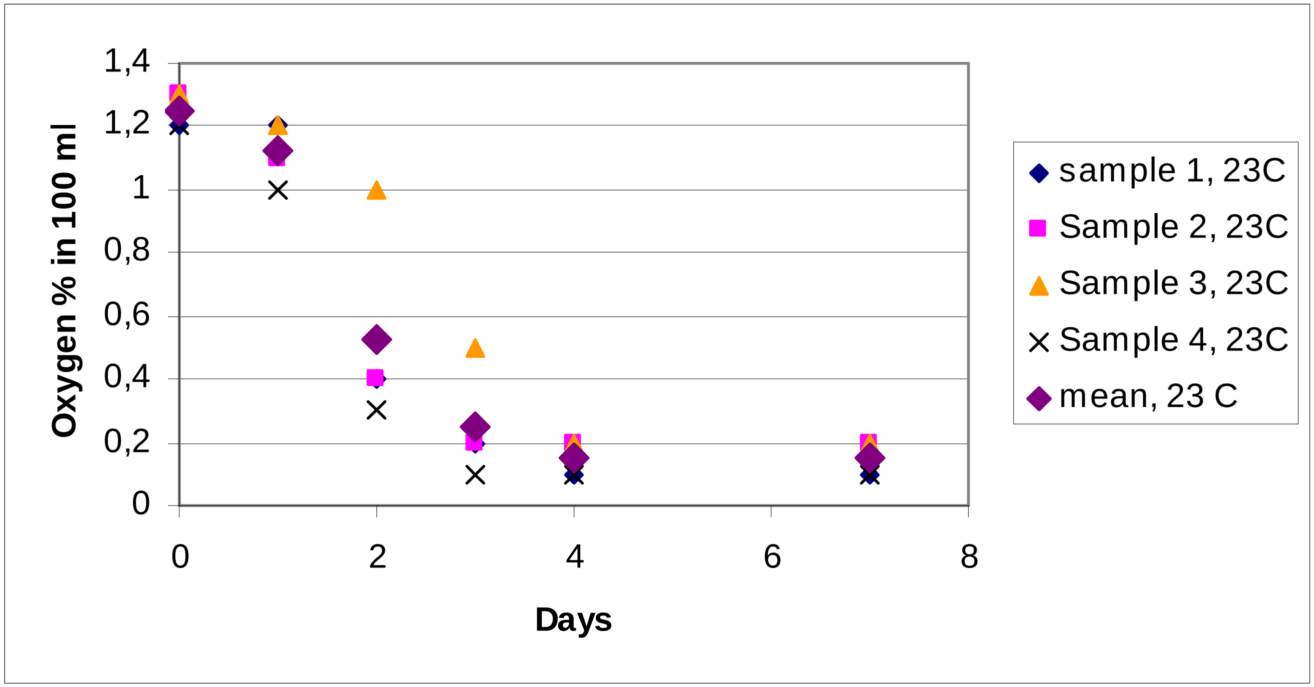
The OxySense utilizes an optical methodology that determines the oxygen concentration within a sealed package by measuring the florescence generated upon illumination of an oxygen-sensitive film (OxyDot®). The fluorescence is proportional to the oxygen concentration in the sealed package.

Instead of testing of packages produced from the enzyme coated board, stripes of the coated board (Enzysheets) were tested in a sealed glass jar (100 ml).

Activation step needed (liquid water).
No reduction in oxygen concentration if the dry (not activated) coated samples were tested.

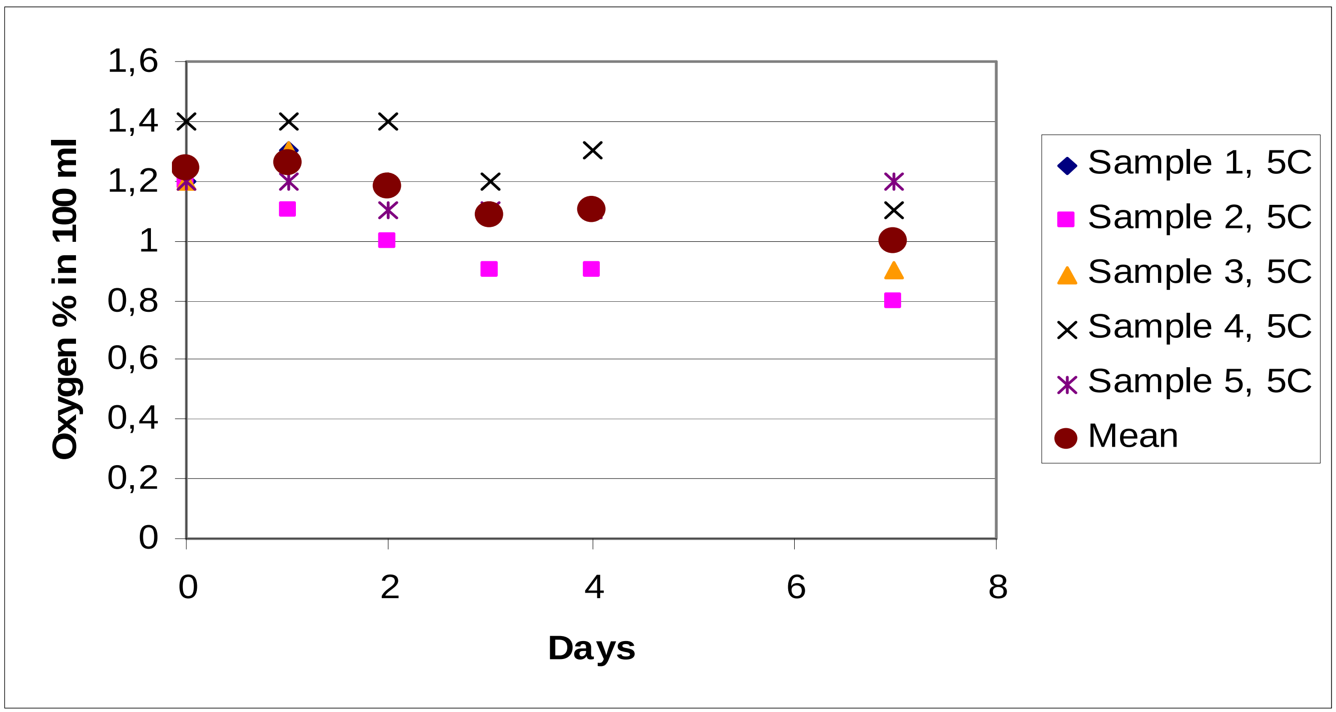
Effect of temperature (I)

Enzysheet activated with liquid water at 23°C for 100 cm^2 of material. The glass jar has a volume of 100 ml. The oxygen level is measured with OxySense.



Effect of temperature (II)

Enzysheet activated with liquid water at 5°C for 100 cm^2 of material. The glass jar has a volume of 100 ml. The oxygen level is measured with OxySense.



The results indicated also that Enzysheets were efficient in reducing the oxygen concentration from high initial levels.

For 8.8 % oxygen in start level: Within 32 days, the oxygen level was decreased to below 3% oxygen. A single sample reached 0.1% oxygen within 9 days.

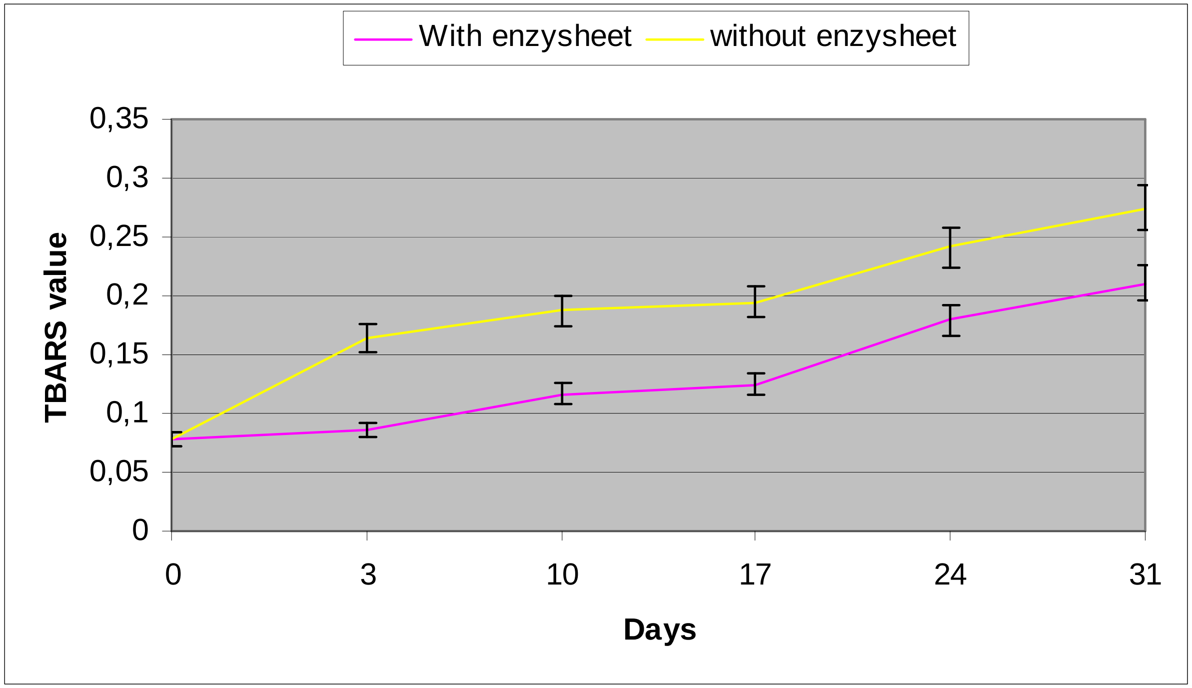
For 23% oxygen in start level: Within 32 days, the oxygen level was decreased until approximately 14 % for 4 samples and until 3% for a single sample.

However, large experimental scatter was observed and additional experiments are needed.

Results:
Rancidity test of cake and minced ham

Rancidity test of minced ham with activated Enzysheet

TBARS value is a test of malondialdehyde, which is a product of the oxidation occurring in the meat. Stored at room temperature and with a 100W light shining on the samples the whole time.



Enzysheets protected the food against oxidation and slowed down rancidity processes.

Results for cakes indicated no effects on rancidity. The Enzysheets could not retard the oxidation taking place in the cakes more than control samples without enzymes (23 and 37 °C).

This shows that additional research is needed regarding

- Food types
- Activation
- Immobilisation
- Mobility of enzymes and substrate molecules
- Inorganic nanostructures or microcapsules as carriers of the enzyme
- etc…

Results:
Heat sealabilty

The coating without TiO_2 is heatsealable.

However even at high sealing temperatures, times and pressure, it produce what could be a hermetic seal, but too weak to be of practical packaging use.

When TiO_2 was included no seal was obtainable.

Conclusions

1. Enzymes embedded in latex films applied in ordinary paper coating or printing processes can retain its activity during long period of times. The enzymes could resist elevated temperatures during coating and drying operations at short time scales.
2. For reduction of oxygen in ambient atmosphere an activation process with liquid water was needed. The samples without activation with liquid water did not absorb oxygen.
3. The Enzysheet was proved to be able to decrease the oxygen level dramatically from a very high start concentration of oxygen.
4. Enzysheets did reduce the oxidation (rancidity) of packed minced pork meat.
5. The coating without TiO_2 is heat-sealable but improvements regarding seal strength are needed.

Acknowledgements

The financial support from the Nordic Innovation Centre (NICe) is gratefully acknowledged.

Thank you for your attention!

I would be happy to answer any questions.