POTENTIALITIES OF COTTONSEED PROTEIN CONCENTRATE IN THE CONTROLLED RELEASE OF INSECTICIDE

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SUMMARY :

THE CONTROLLED RELEASE

SOME GENERAL ASPECTS

POLYMERS AND CONTROLLED RELEASE

The controlled delivery of an active agent happens when it is judiciously combined with a synthetic or natural polymer in such a manner that it is released from the material in a predesigned way

MAIN APPLICATION FIELDS

Agriculture : active seedcoatings
Medicine : drug delivery systems

MAIN ADVANTAGES/OBJECTIVES

“The right dose at the right time and the right place”
Avoid under- or overdosing, increase stability
Reduce amounts and enhance efficiency

RELEASE MECHANISMS

In a given delivery system, the release of an active agent could be ruled by one or a combination of three basic mechanisms

(1) Diffusion through a polymer of stable structure

- macroscopic scale : through matrix pores
- molecular level : between macromolecular chains

(2) Polymer breakdown (case of biodegradable polymers)

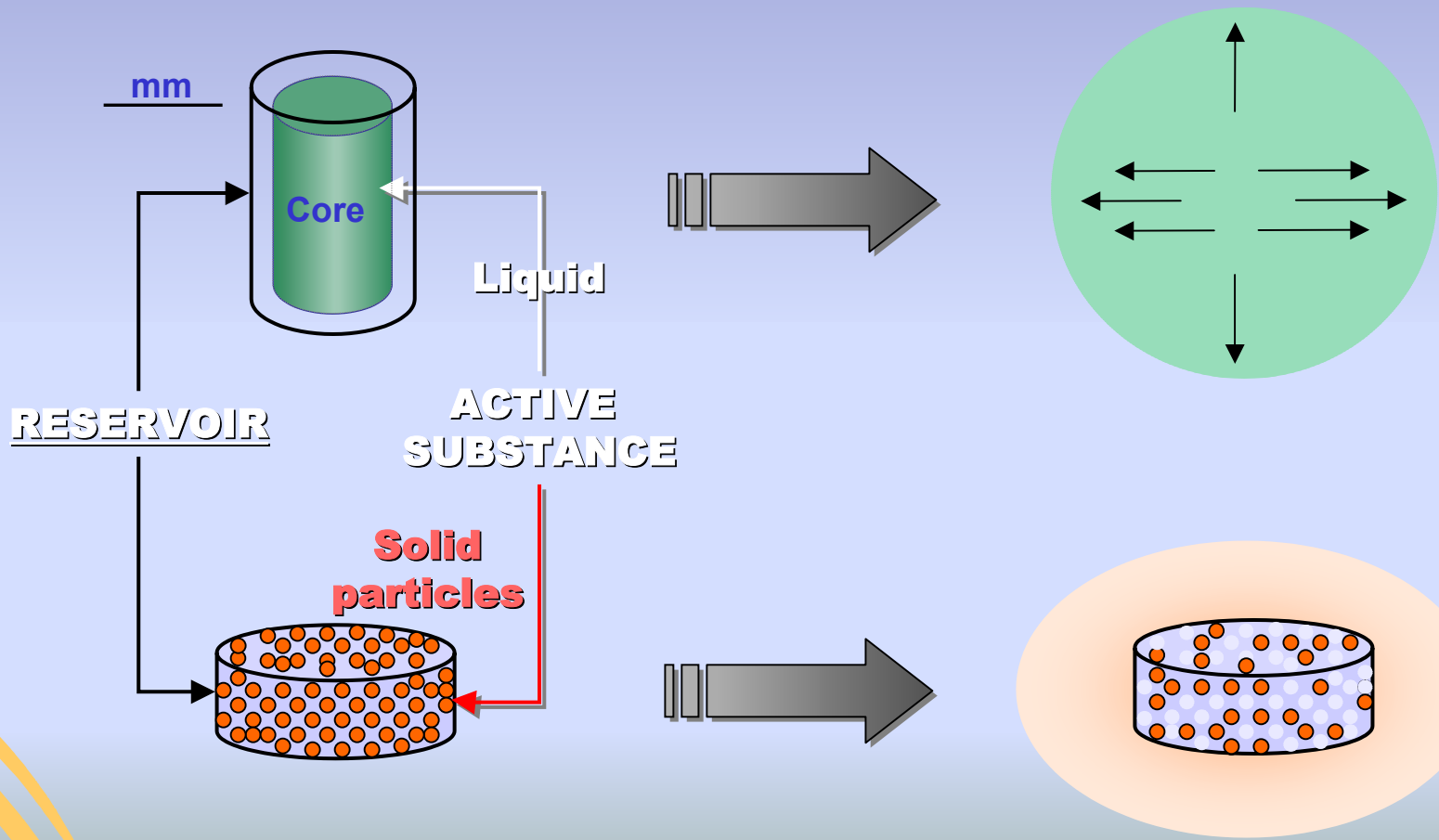
- Bulk erosion
- Surface erosion

(3) Swelling of the matrix and diffusion/Relaxation (case of hydrogels)

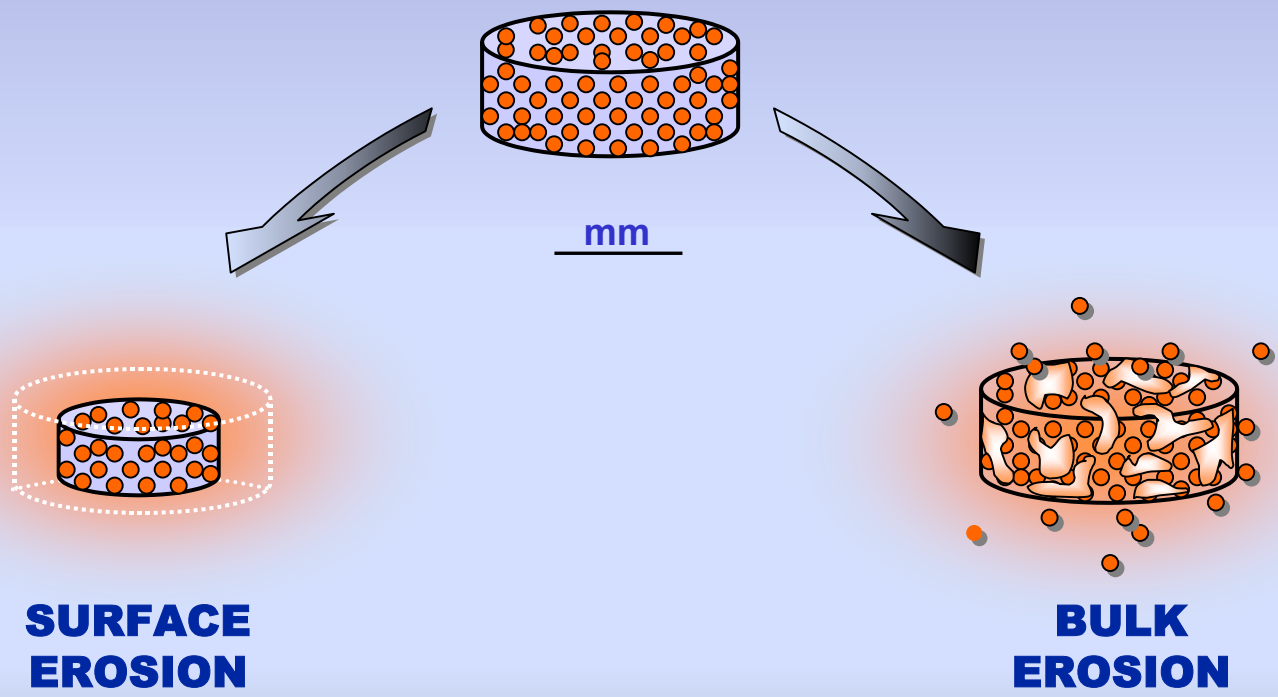
- characterized by $0.5 < n < 1$ (anomalous transport)

In the Ritger-Peppas's equation : fraction released $= k \cdot t^n$

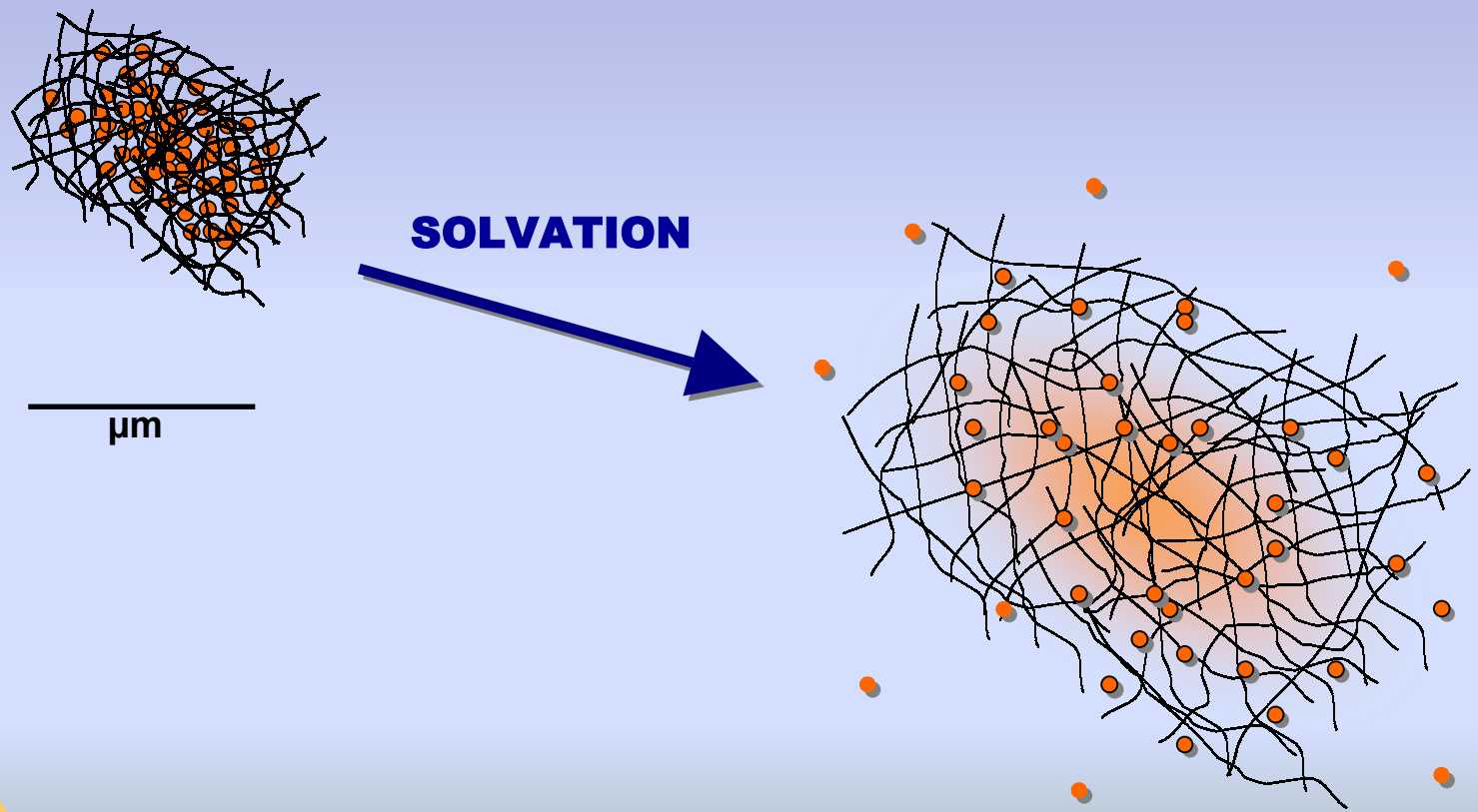
Mechanism 1 :
Diffusion through non degradable polymers



Mechanism 2 :
Release by polymer erosion



Mechanism 3 :
Swelling, relaxation and diffusion



STUDY OF IMIDACLOPRID RELEASE FROM COTTONSEED PROTEINS-BASED MATERIALS

AIM OF THE WORK :

Determine if a cottonseed protein extract used as binder in seedcoating formulations, could control the release of Imidacloprid (Active agent of Gaucho®/ Bayer)

HYPOTHESES :

1) **Protein matrix = Reservoir of active agent**
2) **Influence of cross-linking and plasticization on release**

PRINCIPLE OF THE STUDY :

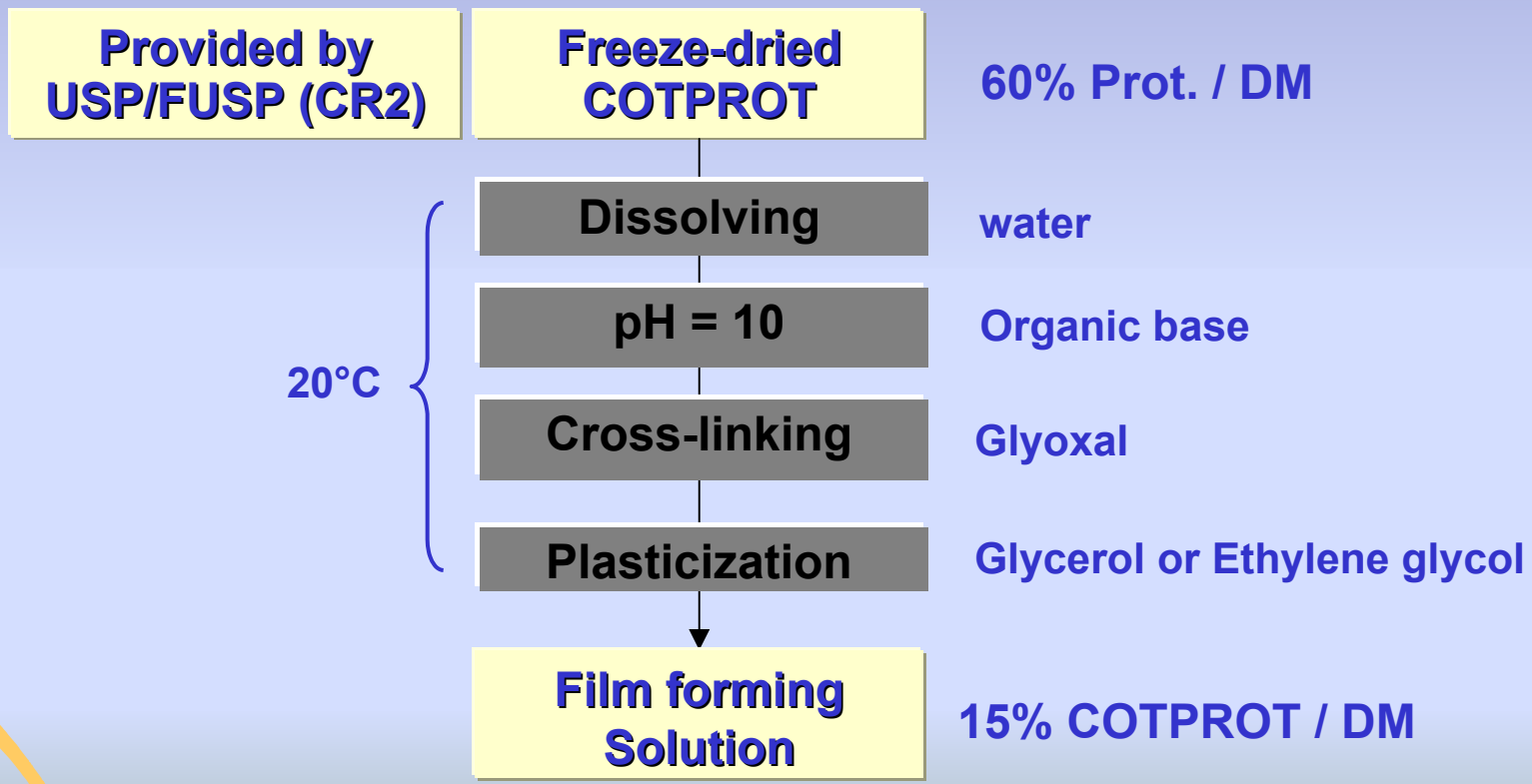
Kinetic studies of Imidacloprid release in water from films obtained in various conditions

PARAMETERS OF THE STUDY

In agreement with INCOTEC BV (CR3, WP3), the parameters of the film forming solution and films preparation were fixed in order to :

- Ensure sufficient cross-linking with medium viscosities
- Avoid gelling and microbiological spoilage
- Lead to contrasted release kinetics
- Be compatible with industrial process constraints

OBTAINING OF FILM-FORMING SOLUTIONS FROM FREEZE-DRIED COTPROT



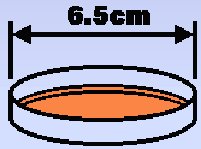
OBTAINING OF COTPROT/ Gaucho® FILMS

Film forming solution

Aqueous Gaucho® solution

Mixing

Pouring in ∅ 6.5 cm dish

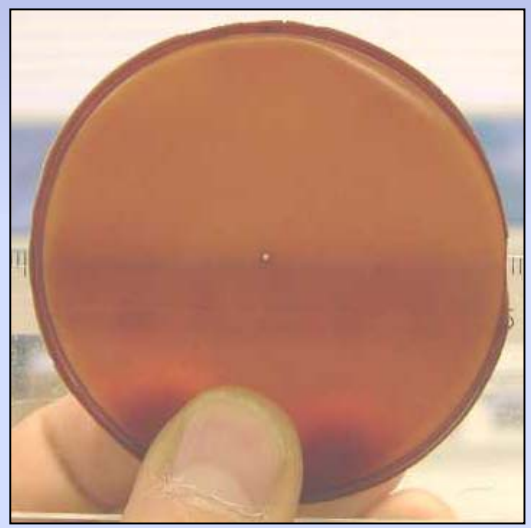


Drying (T_{amb}, air flow)

film

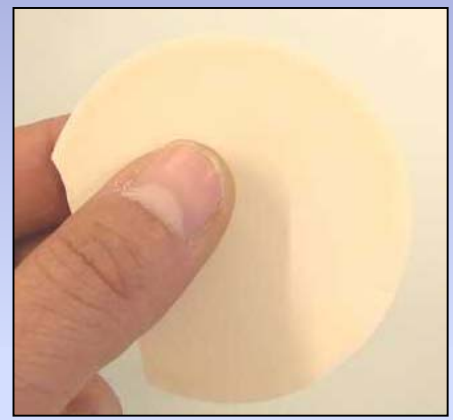
30 mg DM/cm²
Thickness : 200 – 300 µm
Gaucho® content : 55% or 90%

FILMS BEFORE SAMPLING



100%
COTPROT Film

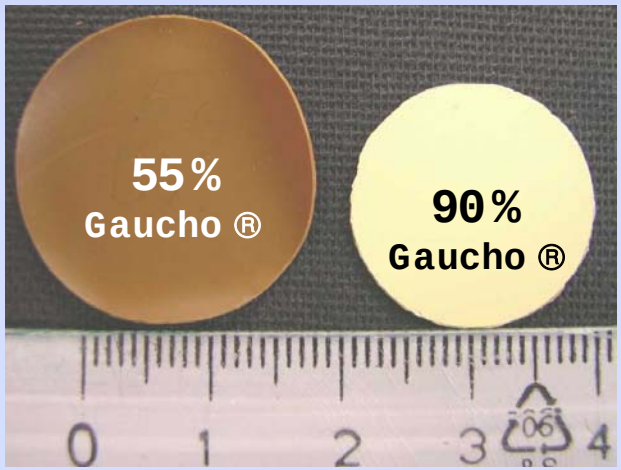
Typical
55% Gaucho® film



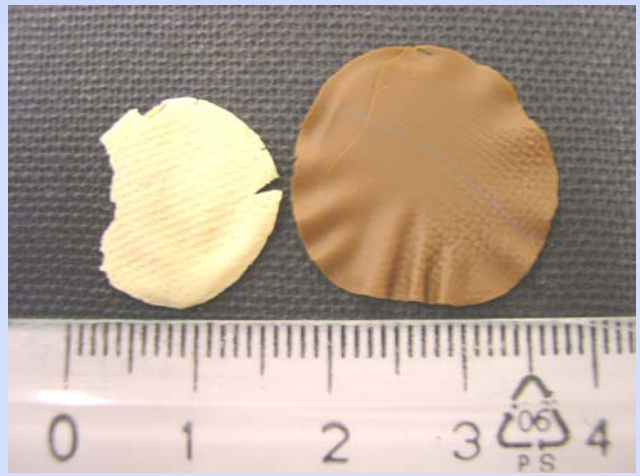
Typical 90%
Gaucho® film

FILMS SAMPLES, BEFORE AND AFTER RELEASE

Films were divided into disc of 20 mm or 25 mm diameter in order to have the same imidacloprid weight, whatever the Gaucho ® content

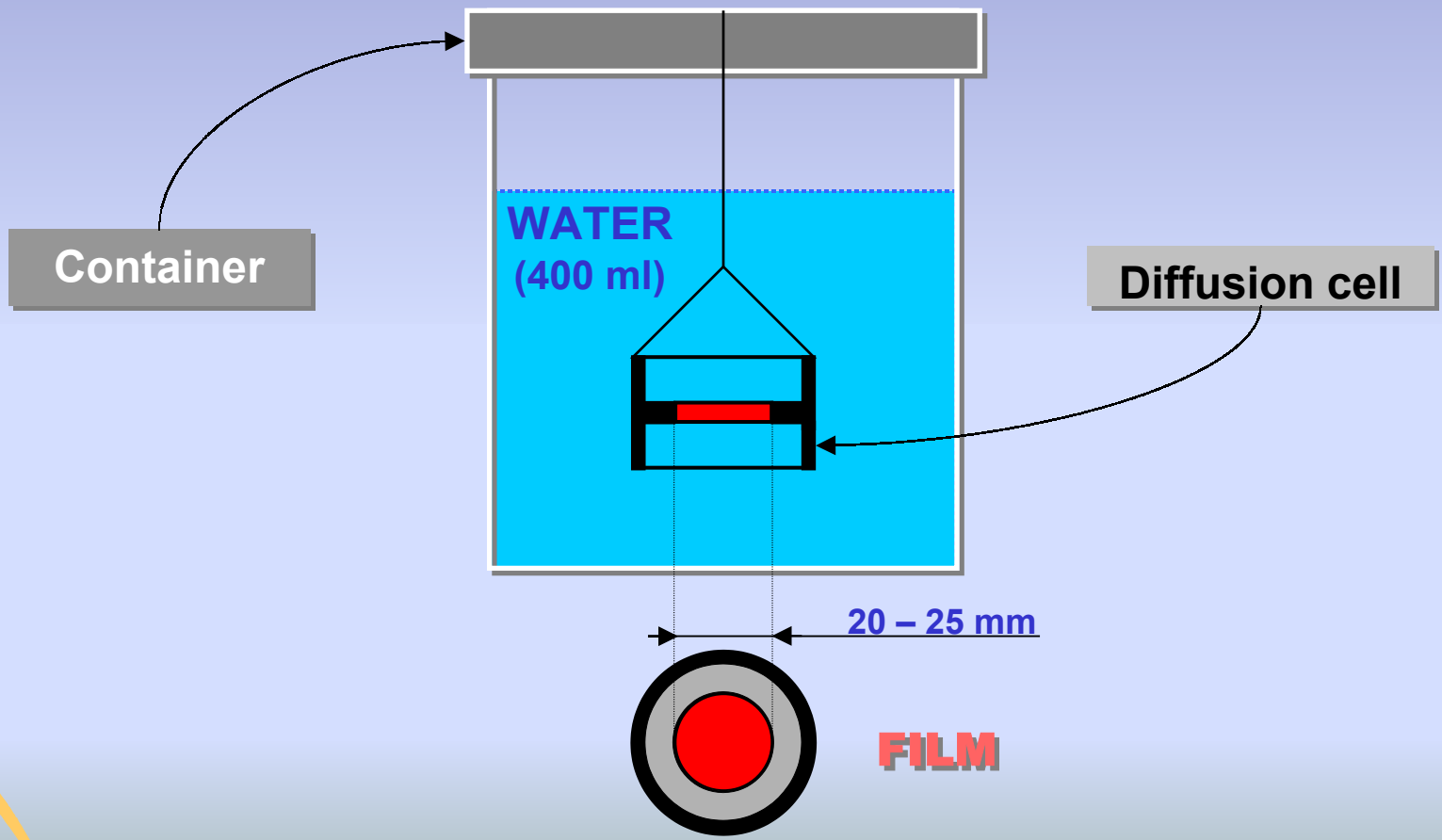


Before release



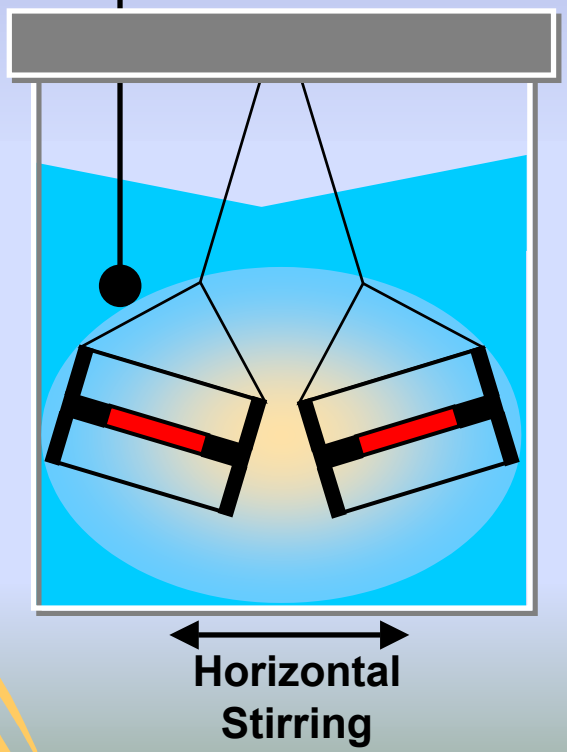
After 30h release

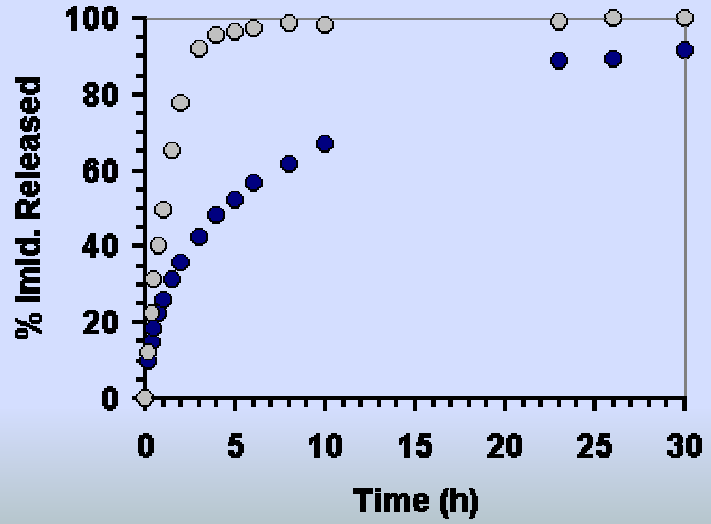
RELEASE DEVICE and KINETICS (1)



RELEASE DEVICE and KINETICS (2)

SAMPLING (30h) → **HPLC ANALYSIS (UV 270 nm)** → **RELEASE KINETICS (2 – 4 replicates/film)**



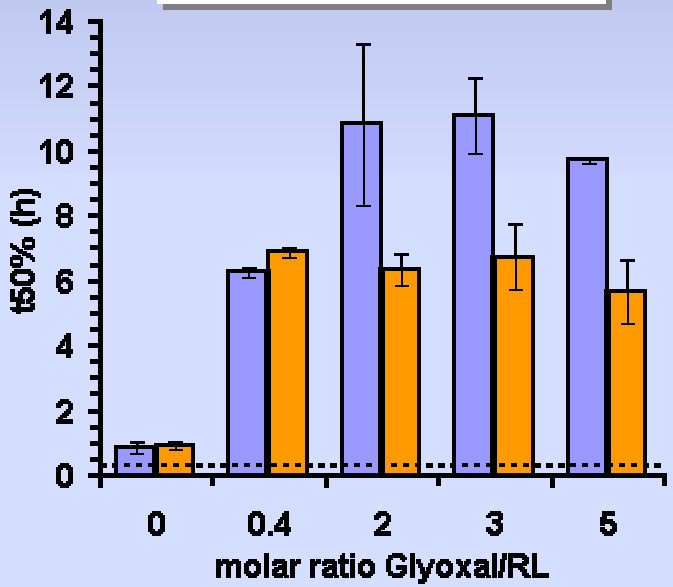


RELEASE TESTS FROM COTTONSEED PROTEINS-BASED FILMS

INFLUENCE OF CROSS-LINKING ON RELEASE RATES

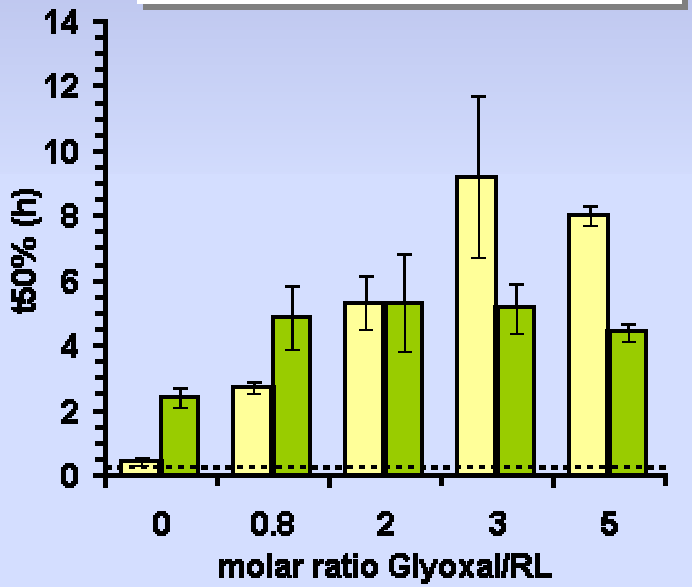
(Comparison of time at 50% Release : $t_{50\%}$)

Plasticizer : GLYCEROL



100% Gaucho® film

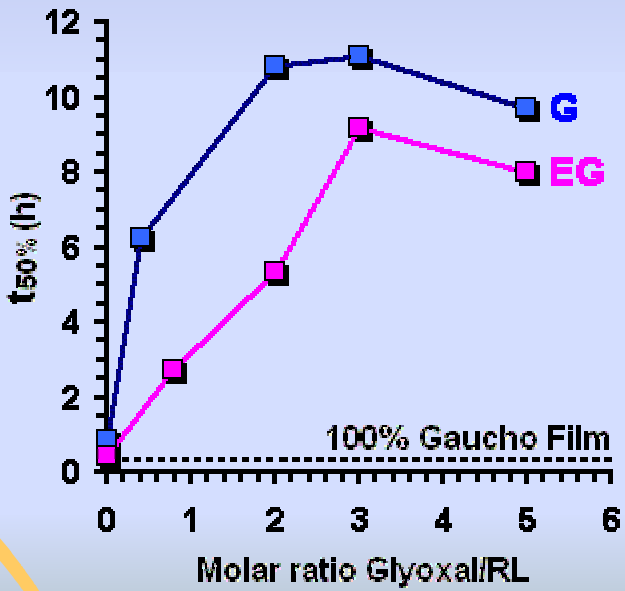
Plasticizer : ETHYLENE GLYCOL



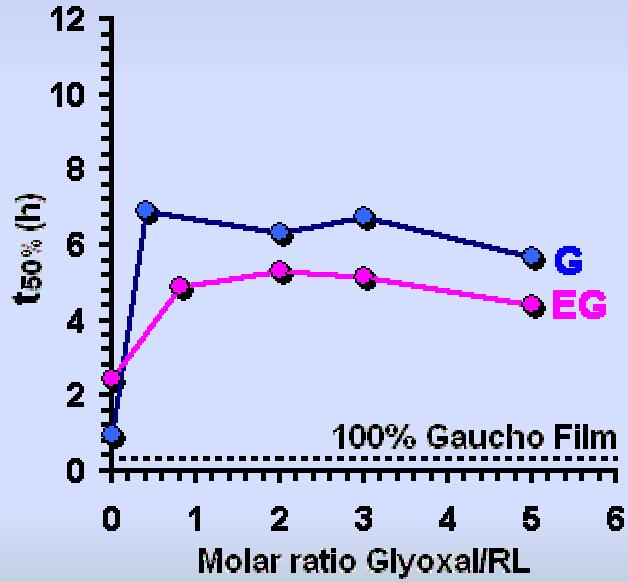
INFLUENCE OF PLASTICIZER TYPE ON RELEASE RATES

(Comparison of time at 50% Release : $t_{50\%}$)

Films of 90% Gaucho®



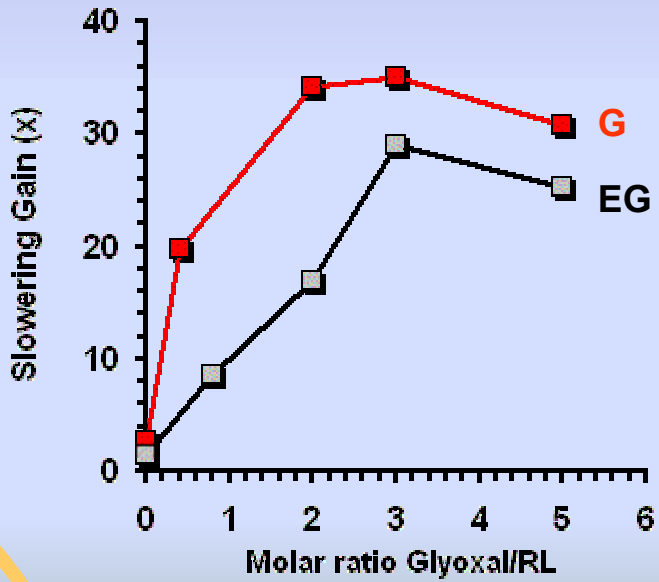
Films of 55% Gaucho®



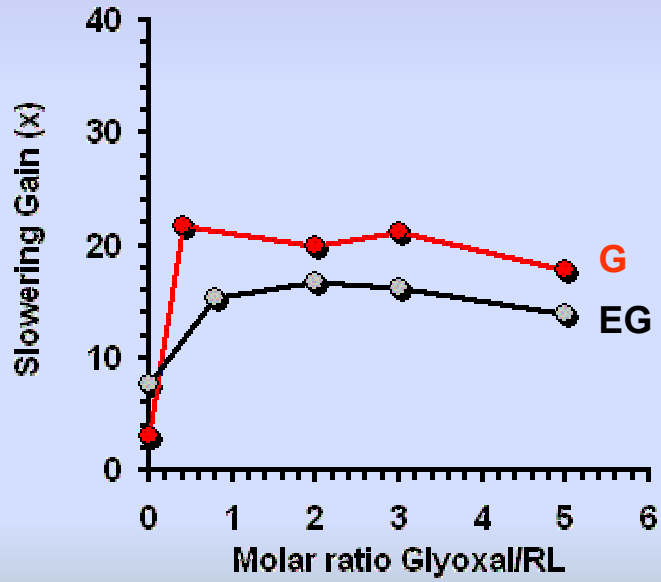
SLOWERING GAINS

(Comparison to a 100% Gaucho film, taken as reference = 1)

Films of 90% Gaucho®



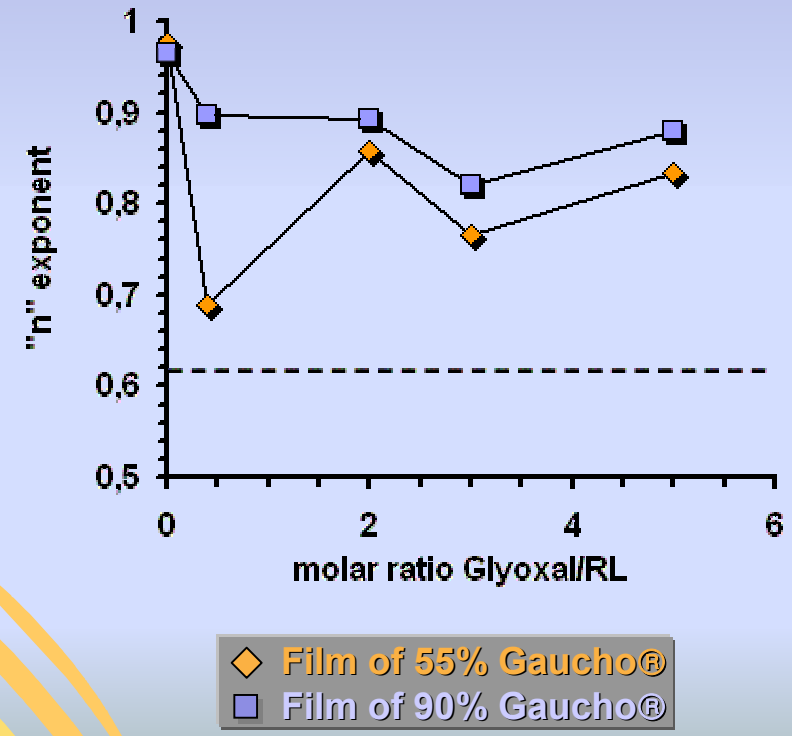
Films of 55% Gaucho®



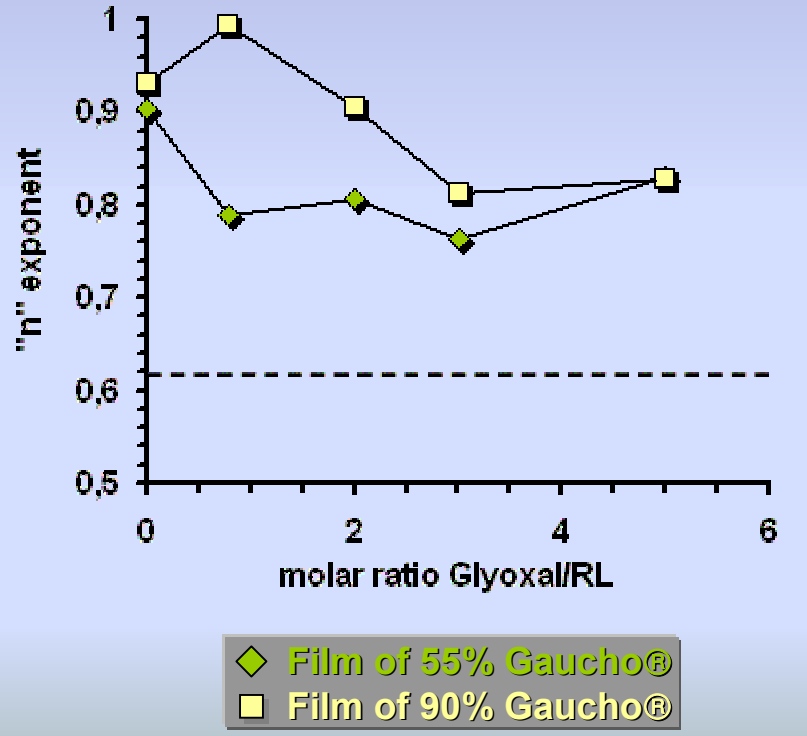
MECHANISM OF RELEASE

Curve Modelling * / Ritger-Peppas's equation : $R\% = k.t^n$

Plast : GLYCEROL



Plast. : ETHYLENE GLYCOL



* CurveExpert software 1.3 by Daniel Hyams

MECHANISM OF RELEASE

Curve Modelling / Ritger-Peppas's equation : $R\% = k.t^n$

Kinetic data modelling :

- $0.7 < n < 1$

Experimental observations :

- Swelling of films in water
- weight loss = initial Gaucho weight
- Recovery of crosslinked films after 30h release (no or low dissolution)

We assumed that the imidacloprid release could be ruled by swelling and relaxation/difffusion phenomena, from films with behaviour similar to hydrogels

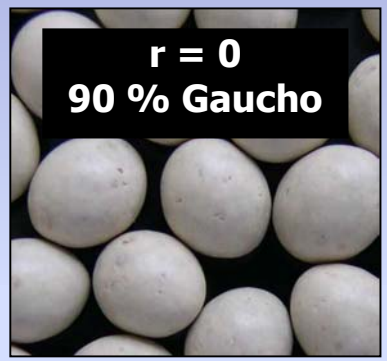
RELEASE TESTS FROM PELLETS

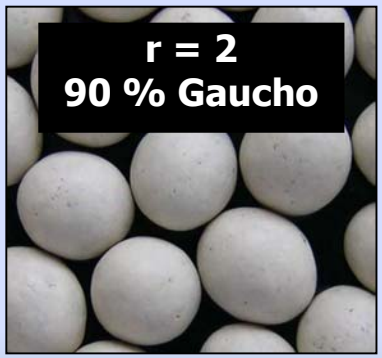
The coating of pelletized seeds was realized at INCOTEC BV (CR3, WP3), according to one of its current coating process

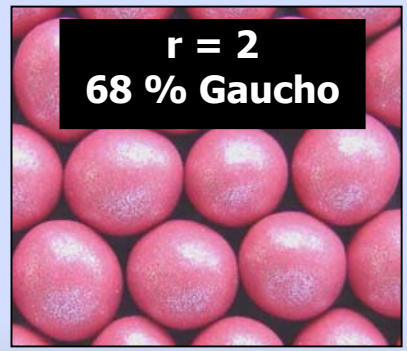
WITH

several film-coatings (FC) containing COTPROT or available commercial binders

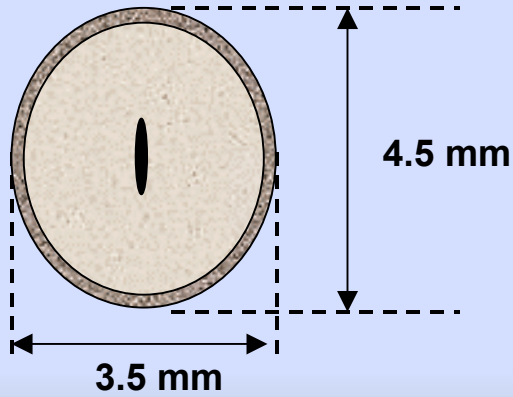
COATED SEEDS WITH COTPROT-BASED FC







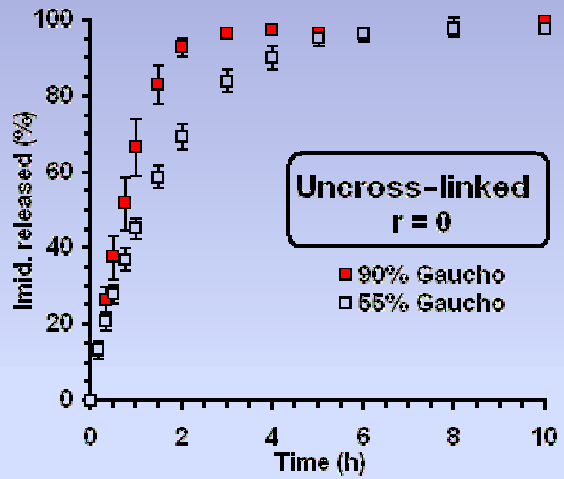
COATED SEEDS WITH INCOTEC's FC

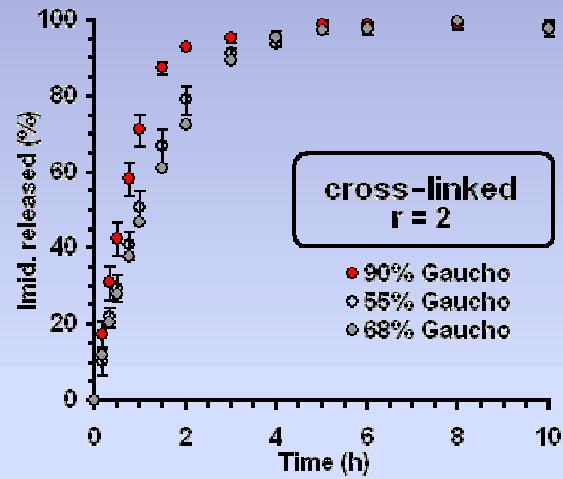


KINETIC RESULTS (1)

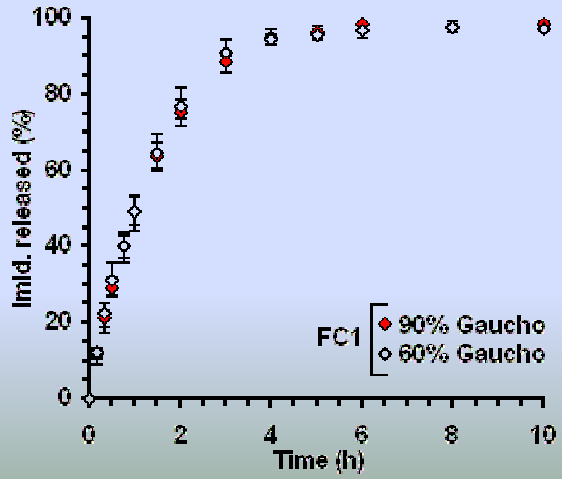
Release kinetic on 40 pellets samples (32 mg imidacloprid)

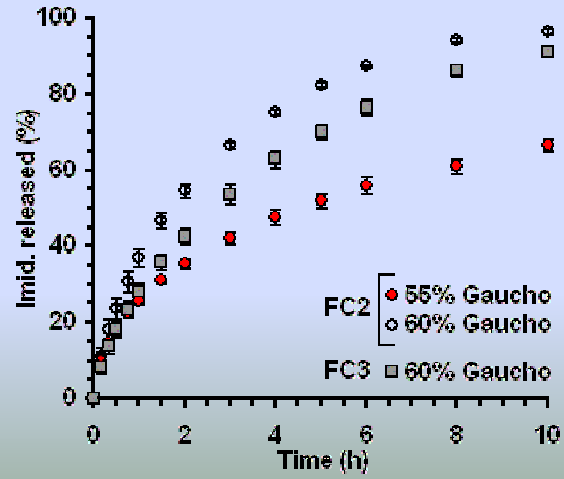
COTPROT FC





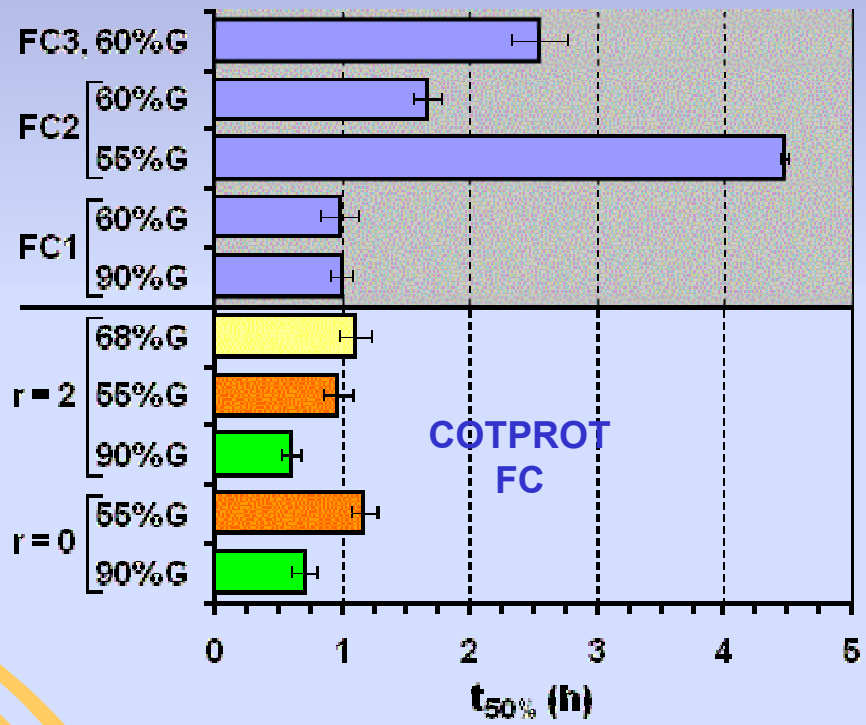
INCOTEC FC

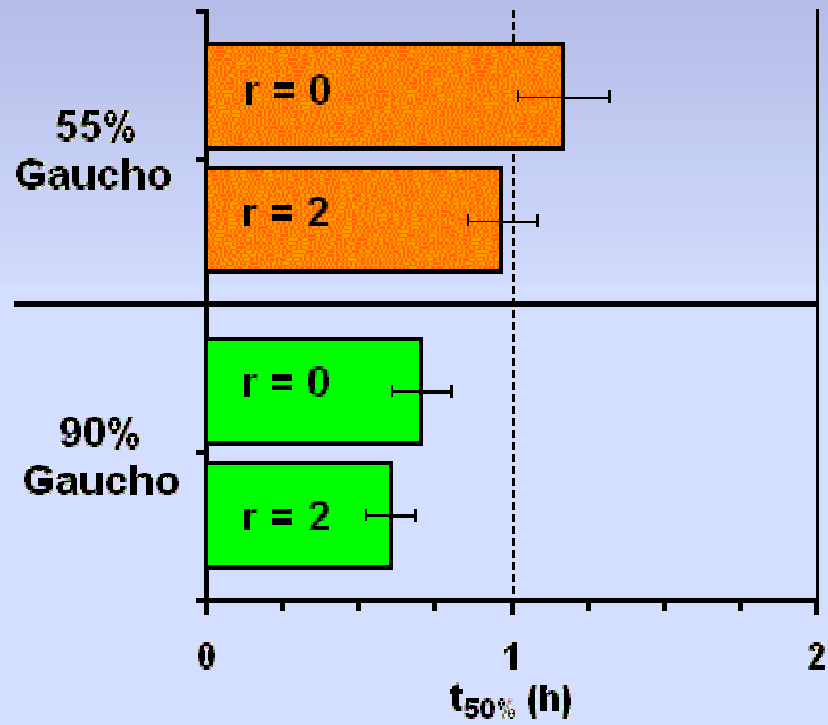




KINETIC RESULTS (2)

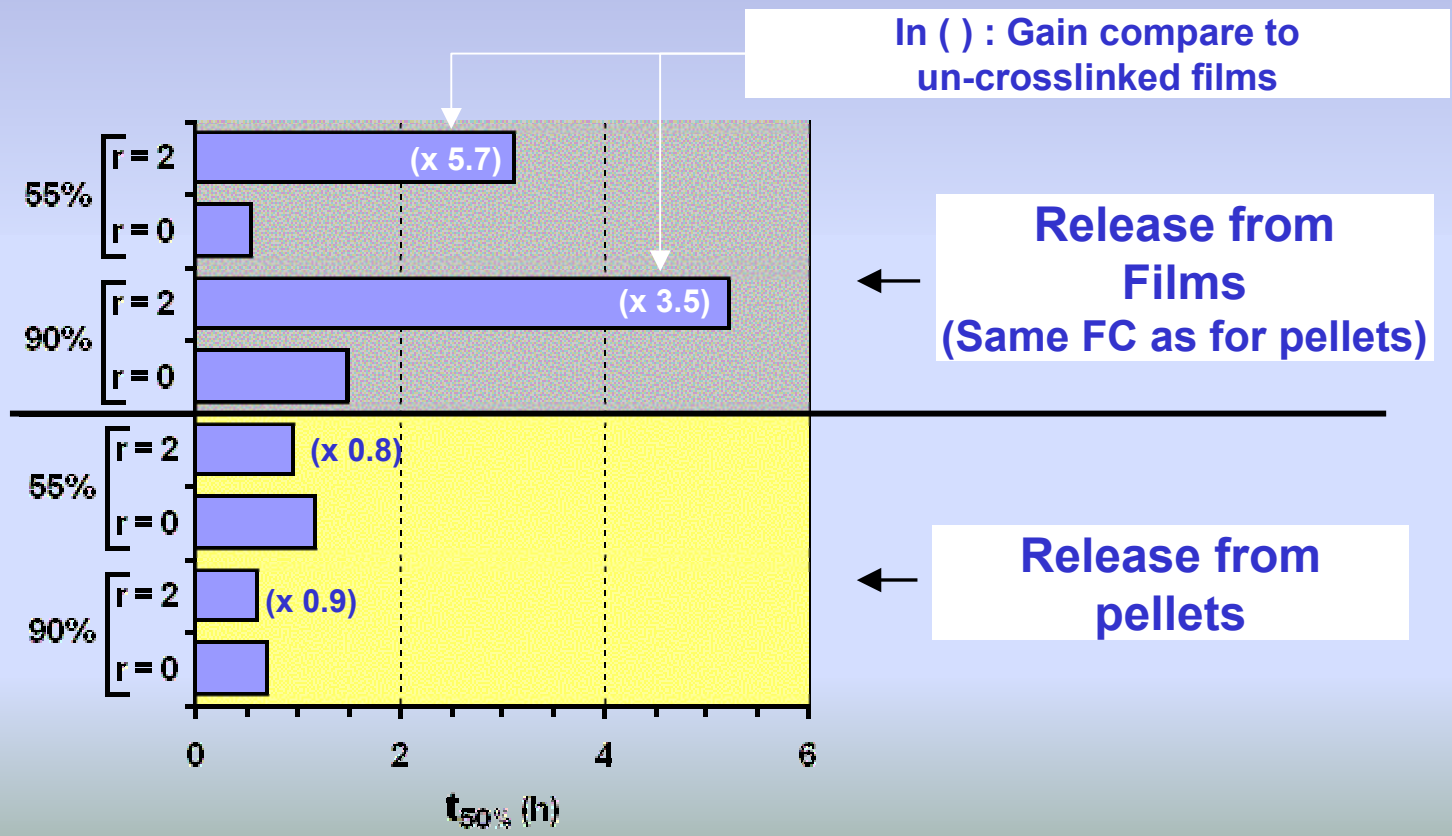
Comparison of time at 50% Release : $t_{50\%}$





KINETIC RESULTS (3)

RELEASE FROM COTPROT FILMCOATINGS
Comparison of films and coated pellets



CONCLUSIONS AND PERSPECTIVES

CONCLUSIONS

SIGNIFICANT REDUCTION OF IMIDACLOPRID RELEASE RATE IN FILMS THANKS TO COTTONSEED PROTEINS

Great influence of :

- Cross-linking (optimimun ratio around 2-3)
- Gaucho content (best results with the highest content)

Slight effect of plasticizer

Slower release with glycerol

UNEXPECTED RESULTS WITH COATED PELLETIZED SEEDS

Fast release and slight influence of cross-linking

PERSPECTIVES

AT LABORATORY SCALE

IMPROVEMENT OF CONTROLLED RELEASE PROPERTIES AND STUDIES OF RELEASE MECHANISMS

- Use of pure cottonseed proteins of specific fractions
- Variation of Hydrophoby/Hydrophily balance of protein (fonctionnalization) and active agents
- Plasticization effect with PEG of various molecular weight
- Influence of film thickness and drying condition
- Study of swelling capacity of films
- Test in normalized conditions

AT INDUSTRIAL SCALE

TEST OF NEW COATING PROCESSES OR ADAPTATION OF CURRENT ONES

ACKNOLEDGEMENTS

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