AEMA ASPHALT EMULSION MANUFACTURERS ASSOCIATION
ibef International Bitumen Emulsion Federation

ISAET '21
7th International Symposium on Asphalt Emulsion Technology

100% Bio Based Emulsifier for Asphalt

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EIFFAGE Routes

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EIFFAGE

Little Introduction

- Deputy Technical Manager for EIFFAGE Route (13 000 people – 2 billion euros Turn Over)
- 51 years old – 23 years in the asphalt industry
- In charge of hydrocarbon binders for the Technical Department
  - PmB
  - Emulsions
  - New Bio binders (anhydrous and emulsions)
- Emulsion based mixes (Cold Asphalt Concrete, Grave Emulsion, CIR etc….)
- Development of eco-friendly emulsifiers

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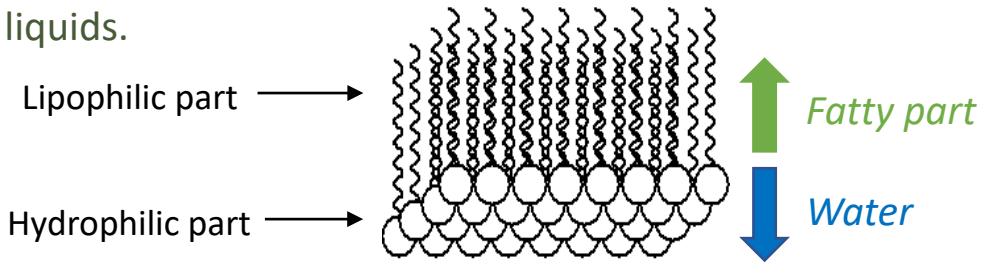
BACKGROUND PRINCIPLES | 1

Background part 1:Typology of an Emulsifier

- **Molecules having the property** to migrate at the interface of two non soluble liquids.



- Properties used **in numerous industrial activities** : cosmetic, drilling et refining of oil, steel industry, agriculture,…
- Molecules initially coming from **petrochemistry**

Limitations : Can be pollutant, toxic for the biomass, not biodegradable.

Rising of **bio based emulsifiers**, being an alternative of conventional chemistry

- **10 %** of the total market but mainly non ionic.
- Yearly growth of **5,6 %** per year with an important share in Europe.
- Mainly non ionic, a few anionic, NOT CATIONIC

Challenges to overcome:

Cost

Lack of diversity

Proven performances ?

Background part 2:Challenges

Emulisifier = Lipophilic chain + hydrophilic head

Sythesis from **vegetable oils**

Fatty Chain	Head	Biosourced
PETROL Based	PETROL Based	0%
Natural based	PETROL Based	~30%
Natural based	**Natural based**	100%

ONLY for non ionic: synthesis from sugars (cellulose, starch, hemicellulose)

There is not any cationic emulsifier that is 90-100% naturally based on the market

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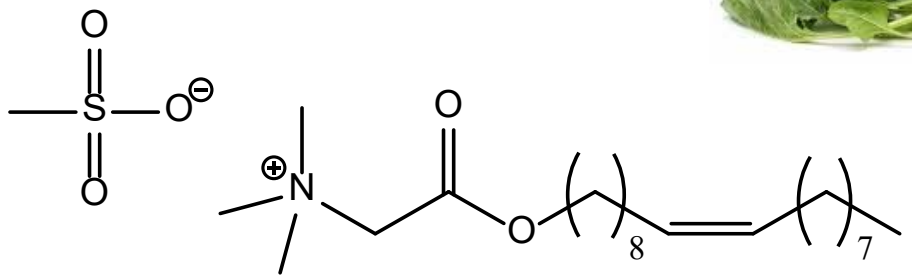
Background part 3: Main Drivers

- Main drivers:
  - Environmental pressure: Less Impact on the environment
  - Societal pressure: Less impact on the health of users and applicators

- Main characteristics :
  - Bio-sourced with renewable raw materials
  - Biodegradable
  - Low toxicity

Inspired by nature..

(Sources from sugar Beets)



Patent WO 2005/121252 A1

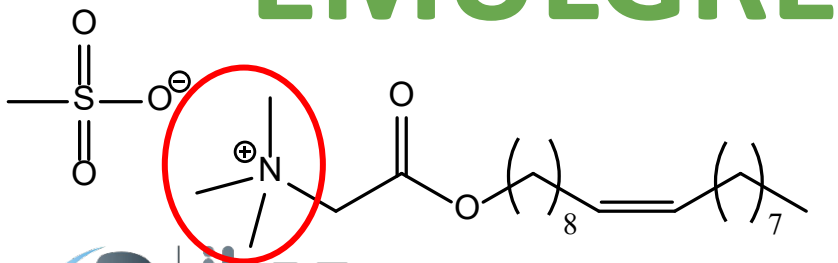
Emulgreen™ R-A: the molecule

According the principles of Green Chemistry

Made in

Oleic Alcohol
Coming from vegetable oils

EMULGREEN ™ R-A



Molecule already activated (cationic)

No need of hydrochloric acid

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PRODUCTION: the 12 principles

Raw and final products must be biodegradable

Production must create very little waste

Segment	E Factor (kg of waste per kg of final product)
Base Chemistry	<1 – 5
Fine chemistry	5 - 50
Pharmaceutical	25 → 100
SurfactGreen	**<0,5**

Atom Economy

The process should allow the maximum incorporation of atoms coming from the raw materials into the final product.

The atom economy in the Surfactgreen™ process is > 95%.

No solvent used

1 batch process

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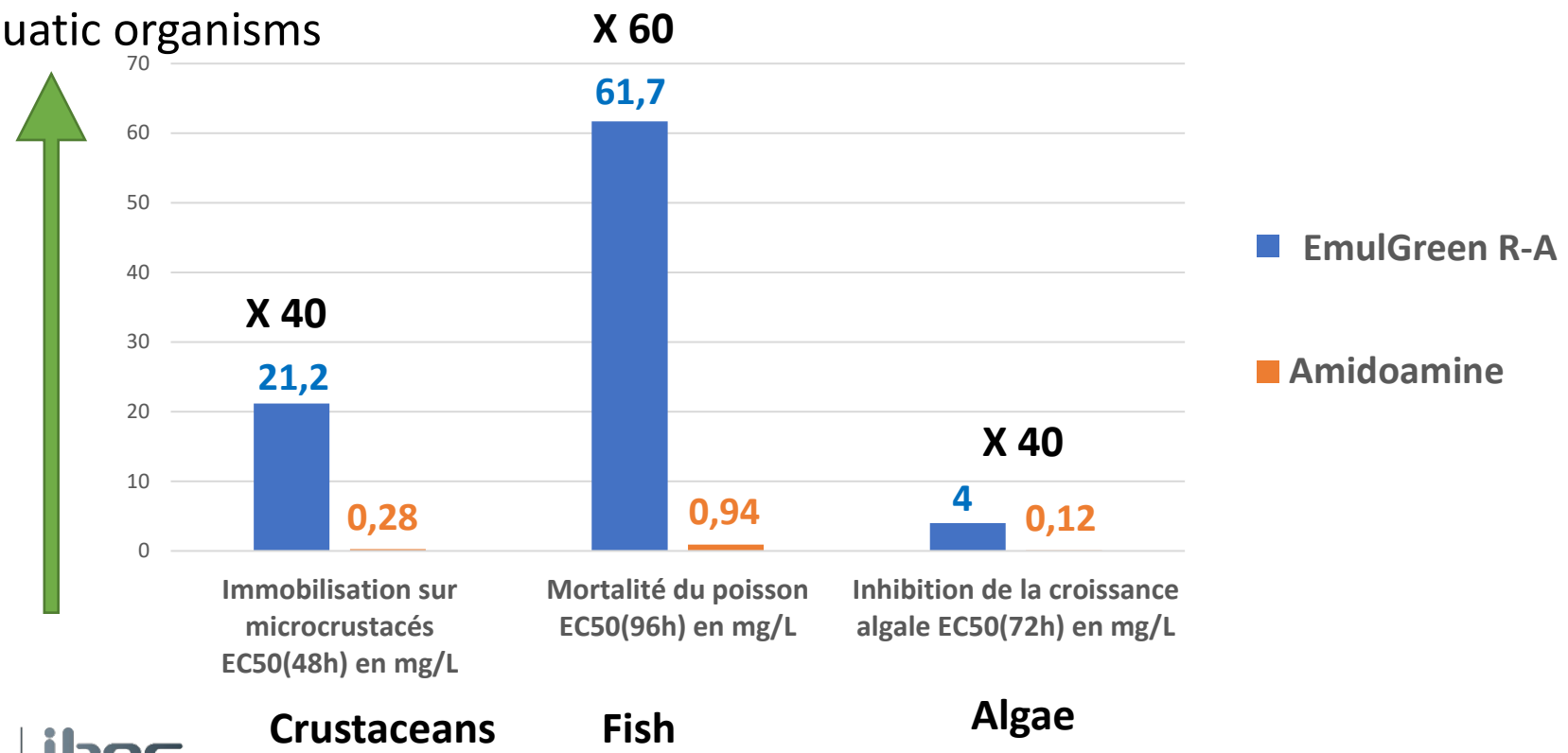
ENVIRONMENTAL IMPACT OF EMULGREEN™

- **Biodegradable** product up to à 94%

(the EN standard stipulates a minimum of 60% to be granted the right of being called « Biodegradable…. »)

- Not Harmful for aquatic organisms

+ the higher
- The less toxic



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CHRONOLOGY EXPERIMENTATION | 2

A long Story…

2004

- Design of « Emulgreen » , cationic rapid set emulsifier based on glycin betain
- Joint Patent with ENSCR/Eiffage

2009

- First trials on roads: validation of the concept

2016

- SurfactGreen SAS is created
- First industrial batches

2018

- Last validation of Emulgreen™ R-A (lab and field)
- Scale up

2019

- First sales – 20 tons/year in Eiffage

Lab Evaluations: complies with NF EN 13-808

Breaking Index (g)

Efflux time (s)

Sieve Residue

Adhesivity (%)

Particle sizing (µm)

Caractéristiques	ActiGreen®R base 70/100 C69 B2	ActiGreen®R base 160/220 C65 B2	ActiGreen®R base 70/100 C65 B2	ActiGreen®R base 160/220 C69 B2
Indices de rupture Forshammer (NF EN 13075-1) Sikaisol	< 110 (2) ≤ 80	< 110 (2) ≤ 80	70-155 (3) ≤ 110	< 110 (2) ≤ 80
Teneur en liant (NF EN 1428) (%)	67 à 71 (9)	63 à 67 (7)	63 à 67 (7)	67 à 71 (9)
Temps d'écoulement (NF EN 12846-1) à 40°C et 2 mm(s) à 40°C et 4 mm(s)	 5 à 70 (5)	40 à 130 (4) -	40 à 130 (4) -	- 5 à 70 (5)
Résidu sur tamis (NF EN 1429) Tamis de 0,500 mm (%) Tamis de 0,160 mm (%) Après 7 jours de stockage, tamis de 0,5 mm (%)	 ≤ 0,1 (2) ≤ 0,25 (2) ≤ 0,5 (4)	 ≤ 0,1 (2) ≤ 0,25 (2) ≤ 0,5 (4)	 ≤ 0,1 (2) ≤ 0,25 (2) ≤ 0,5 (4)	 ≤ 0,1 (2) ≤ 0,25 (2) ≤ 0,5 (4)
Adhésivité passive(NF EN 13614)[1] Sur diorite 6/10 (%)	≥ 75 (2)	≥ 75 (2)	≥ 75 (2)	≥ 75 (2)
Adhésivité active et globale (NF EN 12272-3) sur diorite 6/10 en %	100 100	100 100	100 100	100 100
pH méthode interne	1,5 - 3,5	1,5 - 3,5	1,5 - 3,5	1,5 - 3,5
Diamètre médian (par diffraction optique) en µm	4 à 8 µm	4 à 8 µm	4 à 8 µm	4 à 8 µm

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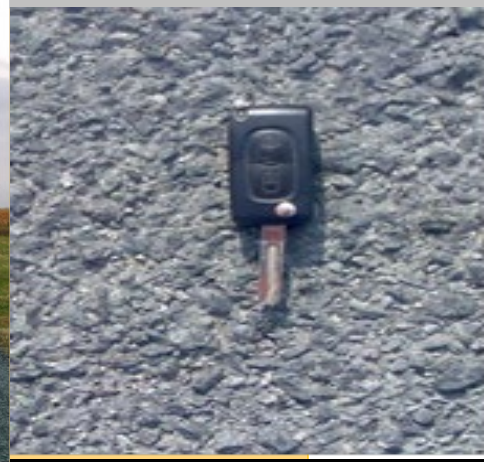
FIELD EVALUATIONS

- **First experimentation in 2009**
  - *Located in France (rural area)*
  - *Traffic cat. (T4) : 25 to 50 trucks / day*
  - *Surface = 16 000 m²*

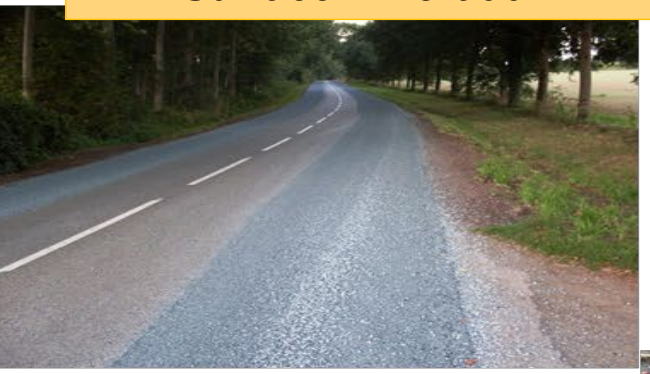
- **Double prechipped surface dressing**
  - *Pure bitumen emulsion*
  - *Bitumen content : 65%*
  - *Layers emulsion content : 1,8 & 1,9 kg/m²*
  - *Agregates (2/4 - 6/10 – 10/14) : ~8 l/m²*



FIELD EVALUATIONS

- **Last experimentations in 2018 (Scale up samples)**
  - *2 worksites (rural area)*
  - *Located in France*
  - *Traffic cat. (T3$^-$) : 50 to 85 trucks / day*
  - *Surface = 25 000 m²*

- **Double layer surface dressing**
  - *Pure bitumen emulsion*
  - *Bitumen content : 65%*
  - *Layers emulsion content : 1,4 & 1,1 kg/m²*
  - *Aggregates (6/10 – 4/6) : 7 & 5 l/m²*



FIEL EVALUATIONS on scale up samples of EMULGREEN R-A ™

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Comparative testing of two surface dressing portions with EMULGREEN R-A ™ and a conventional (petrol based) emulsifier

- No structural defect observed
- Same ease of application –
- Same behavior on site. Same adhesion on aggregates. Very little chip loss.
- Durability validated after a 3 year screening on site.

Decision was made to incorporate EMULGREEN R-A in the emulsion design of tack coats and certain surface dressing

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CONCLUSION PERSPECTIVES | 3

CONCLUSION - PERSPECTIVES

- IT took **fifteen years** from the patent to the general use of the product in our emulsions…
- Needed to find an industrial partner: Eiffage is not a chemical company
- Mentality and societal pressure changed during this time and were not so strong in 2005
- BUT only 2 YEARS from the first real scaled up batches to the validation and generalization of the product by EIFFAGE (2018-2019)
- Since then, EIFFAGE uses about 20 tons of this GREEN emulsifier every year

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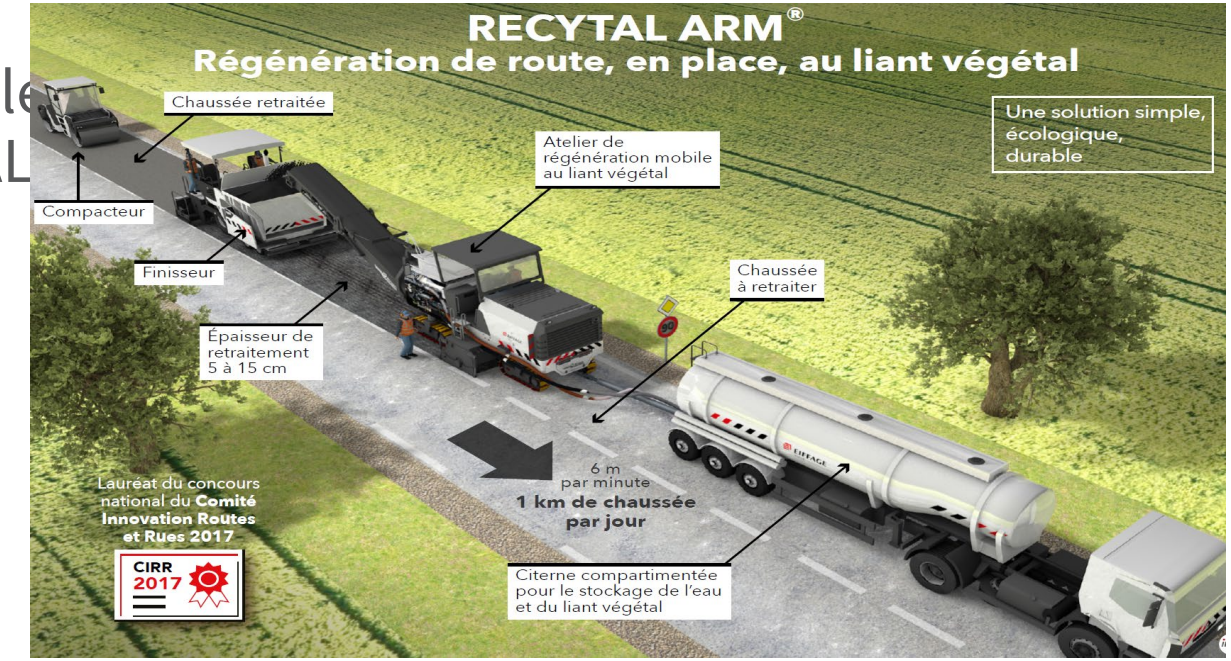
PERSPECTIVES

- A two year research program has been engaged with SURFACTGREEN
- 2 main axes:
  - Optimization of the cost and ease of use of Emulgreen™ R-A (paste to liquid emulsifier)
  - Development of slow setting molecule
    tech RECYTAL



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THANK YOU FOR YOUR ATTENTION

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