



# AgChem Additives by BASF

## Oil Dispersions (ODs)

May 2024

# Agenda

- 1 | Introduction
- 2 | BASF additives for OD
- 3 | OD testing
- 4 | Examples, trouble shooting



# Formulation types:

## Active ingredient physchem properties define formulation type



a.i. is water-soluble

### SL – Soluble Concentrate

a.i. dissolved in water (clear to opalescent liquid) e.g. Glyphosate



a.i. is water-insoluble

### EC – Emulsifiable Concentrate

a.i. is water-insoluble and dissolved in hydrophobic solvents. Emulsifiers are added to formulation to enable spontaneous emulsification upon dilution in water, a.i. remains in the solvent/oil.



### SC – Suspension Concentrate

A high concentration, stable suspension of finely dispersed a.i.(s) particles in water.



### OD – Oil Dispersion

a.i. is insoluble in both oil and water and dispersed as small particles in oil. In the spray solution, a suspo-emulsion is formed.



# OD compared to other formulation types

## ■ Benefits

- ▶ Suitable for water-sensitive / hydrolytically unstable a.i.(s)
- ▶ Base oil may improve leaf penetration of a.i. (adjuvancy)

## ■ Compared to EC

- ▶ Either no suitable solvent available or solvent leads to a.i. degradation
- ▶ EC solvents and base oils of OD formulations can improve uptake

## ■ Compared to SC

- ▶ Some a.i.s rapidly degrade in water and cannot be used in an SC
- ▶ Preparation of an OD similar to SC

## ■ Compared to WG

- ▶ WG is a safer formulation type for hydrolysis sensitive a.i.s but more expensive
- ▶ WG often does not allow incorporation of sufficient amount of adjuvants

# Oil Dispersion (OD)

## Requirements to formulation

### ■ Formulation

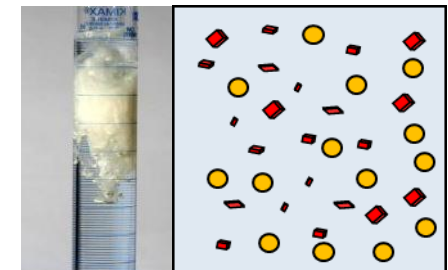
- ▶ Appearance
- ▶ Viscosity, aim for 200 – 800 mPa·s (Brookfield, spindle 3, 30 rpm)
- ▶ Stability i.e. no sediment and no gelling

### ■ Spray solution

- ▶ OD with low viscosity and thixotropic properties
- ▶ Easy to rinse from container
- ▶ Spontaneous dispersibility/emulsification in hard and soft water
- ▶ Stable emulsions with minimal creaming and separation



Dilution

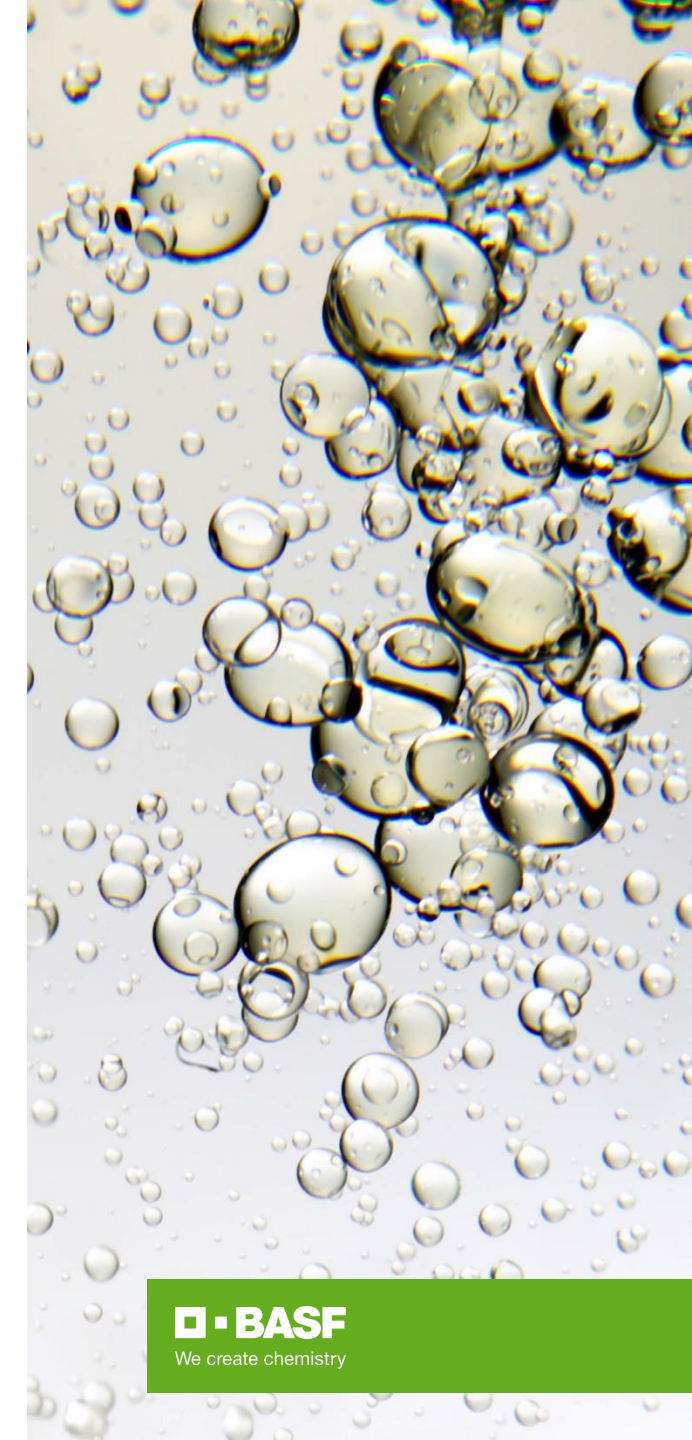


# Generic OD formulation

Function	Content (g/L)	Comment
Active ingredient	40 – 600	Typically 1 – 3 a.i.s., insoluble in base oil to avoid crystal growth. Melting point of a.i. > 70 °C
Base oil	200 – 800	Water-insoluble oil. Can enhance penetration into leaves (adjuvancy)
Non-ionic emulsifier	60 – 80	Non-ionics emulsifiers preferred, optionally methyl-capped
Anionic emulsifier	0 – 50	If required, in combination with non-ionic emulsifier
Adjuvant	0 – 200	Only required if base oil does not provide sufficient adjuvancy
Oil dispersant	0 – 70	To ease milling and to reduce viscosity at elevated a.i. concentrations.
Aqueous dispersant	0 - 70	In the spray tank mix, controls flocculation
Rheology agent	30 - 50	e.g. fumed silica, bentonites
Stabilizer	0 – 25	To control oxidation(if required)

# Most popular OD formulations in Europe

Active(s)	Indication
Nicosulfuron	Herbicide
Oxathiapiprolin	Insecticide
Mesotrione / Nicosulfuron	Herbicide
Mesotrione/ Pyridate	Herbicide
Cyantranilprole	Insecticide
Tembotrione	Herbicide
Spirotetramat	Insecticide
Iodosulfuron-M / Mesosulfuron-M	Herbicide
Foramsulfuron / Thiencarbazon-M	Herbicide
Nicosulfuron / Sulcotrione	Herbicide
Beauveria bassiana	Insecticide
Florasulam / Halauxifen-methyl	Herbicide
Prohexadione-Calcium	Plant growth regulator



# Oil Dispersion (OD)

## Selection of co-formulants

- Co-formulants do not degrade a.i., for example, have minimum aq. content
- **Base oil** can act additionally as penetrants for systemic a.i.(s)
  - ▶ Solvent naphtha, mineral or vegetable oils common, yet, provide minimal penetration of a.i.
  - ▶ Methylated seed oils (e.g. Agnique® ME 18 RD-F) significantly enhance foliar uptake of systemic a.i.s
  - ▶ Agnique® AE 829 is a very hydrophobic oil and a good penetration agent
- **Emulsifiers** and **rheology agent** selection depends on base oil
- **Stabilizers** can be added as water scavenger (e.g. Agnique® ESO 81-G)



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# Product portfolio overview

## Solvents and base oils

Dimethyl amides  
Alkyl & methyl esters  
Carbonates

## Adjuvants

Alkoxyated alcohols  
Alkyl polyglucosides  
Alkyl ether sulfate  
Sulfosuccinate

## Dispersants

EO-PO-EO copolymers  
Polycarboxylates  
Sulfonic acid condensates

We offer products from a **broad technology base** using both petrochemical and renewable raw materials

## Non-ionic emulsifiers

Castor oil ethoxylates  
EO-PO-EO copolymers  
Sorbitol ester ethoxylates

## Ionic emulsifier

Alkyl benzene sulfonates  
Alkyl ether sulfates  
Alkyl sulfates

## Stickers and thickeners

Epoxidised oils  
Polyvinylpyrrolidone homo- and copolymers

## Micronutrients and Chelating agents

EDDHA- and EDTA-based micronutrients  
EDTA

# Sustainability drivers



## Products with bio-based content

Product classification <sup>a)</sup>	Bio-based carbon [X% of total C]	Icon
Wholly bio-based	$X \geq 95$	
Majority bio-based	$95 > X > 50$	
Minority bio-based	$50 \geq X > 5$	
Non bio-based	$X \leq 5$	



## RSPO-certified products

Products available with RSPO mass balance certification



## Readily biodegradability products

Readily biodegradable according to OECD 301<sup>b)</sup>



## Label-free according GHS EU criteria<sup>c)</sup>



## Product with Negative PCF

Product Carbon Footprint (cradle-to-gate)<sup>d)</sup>

- <sup>a)</sup> According to the EN17035 surfactants classification
- <sup>b)</sup> In case of mixture all organic components are readily biodegradable
- <sup>c)</sup> Status of October 2023. Please check the latest MSDS
- <sup>d)</sup> The PCF calculations follow the requirements and guidance given by ISO 14067:2018.



# Additives for OD formulation

## Base oils / stabilizer

Product	Sustainability profile	Chemistry	Function
Agnique® ME 18 SD-F		Soybean oil fatty methyl ester	Base oils. Good penetrators for any fully systemic a.i.s. Proven record with selective herbicides.
Agnique® ME 18 RD-F		Rapeseed oil fatty methyl ester	
Agnique® BL 3095		Emulsifiable methylated rapeseed oil	
Agnique® AE 829		1,2-Cyclohexane dicarboxylic acid, di-isononyl ester	
Agnique® ESO 81-G		Epoxidised soybean oil	Stabilizer in ODs (radical scavenger)








# Agnique® ME 18 RD-F / SD-F

**Sustainable methyl esters** as basis for OD formulations and tank-mix adjuvants

## Phys.-chem. properties

	Agnique® ME 18 SD-F	Agnique® ME 18 RD-F
<b>Chemical structure</b>	Fatty acid methyl esters	
<b>Hansen SP <math>\delta_d/\delta_p/\delta_h</math> (MPas<sup>1/2</sup>)</b>	16.4 / 4.3 / 4.1	16.2 / 3.5 / 4.1
<b>Density 25°C (kg/l)</b>	0.88	0.88
<b>Boiling point (°C)</b>	approx. 355	approx. 355
<b>Solubility in water (g/L)</b>	Insoluble	Insoluble
<b>Flash point (°C)</b>	> 100	174
<b>Freezing point (°C)</b>	< - 5	< -10

## Sustainability

-  Enhance biological performance of crop protection formulations
-  Label-free according to GHS criteria<sup>a)</sup>
-  Majority bio-based product<sup>b)</sup>  
→ bio-based carbon content between 50-95%
-  Readily biodegradable according to OECD 301
-  Product with negative PCF<sup>c)</sup>  
Product Carbon Footprint (cradle-to-gate)

<sup>a)</sup> Status of October 2023. Please check the latest MSDS

<sup>b)</sup> According to the EN17035 surfactants classification

<sup>c)</sup> The product carbon footprint (PCF) calculations follow the requirements and guidance given by ISO 14067:2018.

**Enhance uptake of hydrophobic actives with systemic action e.g. selective herbicides**

# Agnique® AE 829

## Hydrophobic base oil for OD formulations improving leaf penetration

### Phys.-chem. properties

Chemical structure	1,2-Cyclohexane dicarboxylic acid, di-isononyl ester
Viscosity 25°C (mPa·s)	55
Density 25°C (kg/L)	0.95
Boiling point (°C)	394
Flash point (°C)	224
Freezing point (°C)	-54
logP* (calc.)	10
Solvent into water (mg/l)	<0.02

### Sustainability

 Enhances biological performance of crop protection formulations

 Label-free according to GHS criteria<sup>a)</sup>

<sup>a)</sup> Status of October 2023. Please check the latest MSDS

### Regulatory status and inventory listing

CAS number <sup>1)</sup>	166412-78-8
China IECSC	Registered
EU REACH <sup>2)</sup>	Registered
USA TSCA	Listed
USA EPA: 40 CFR 180	(not supported)

<sup>1)</sup> CAS number of main component

<sup>2)</sup> REACH registration: The product may be traded inside the European Union (EU), i.e. the ingredients are either registered or exempted

<sup>3)</sup> Listed as active in TSCA inventory

### Adjuvant: Uptake enhancement

#### Simulation of foliar penetration (SOFP) test:

**Active:** <sup>14</sup>C Epoxiconazole















**Cuticle:** Prunus

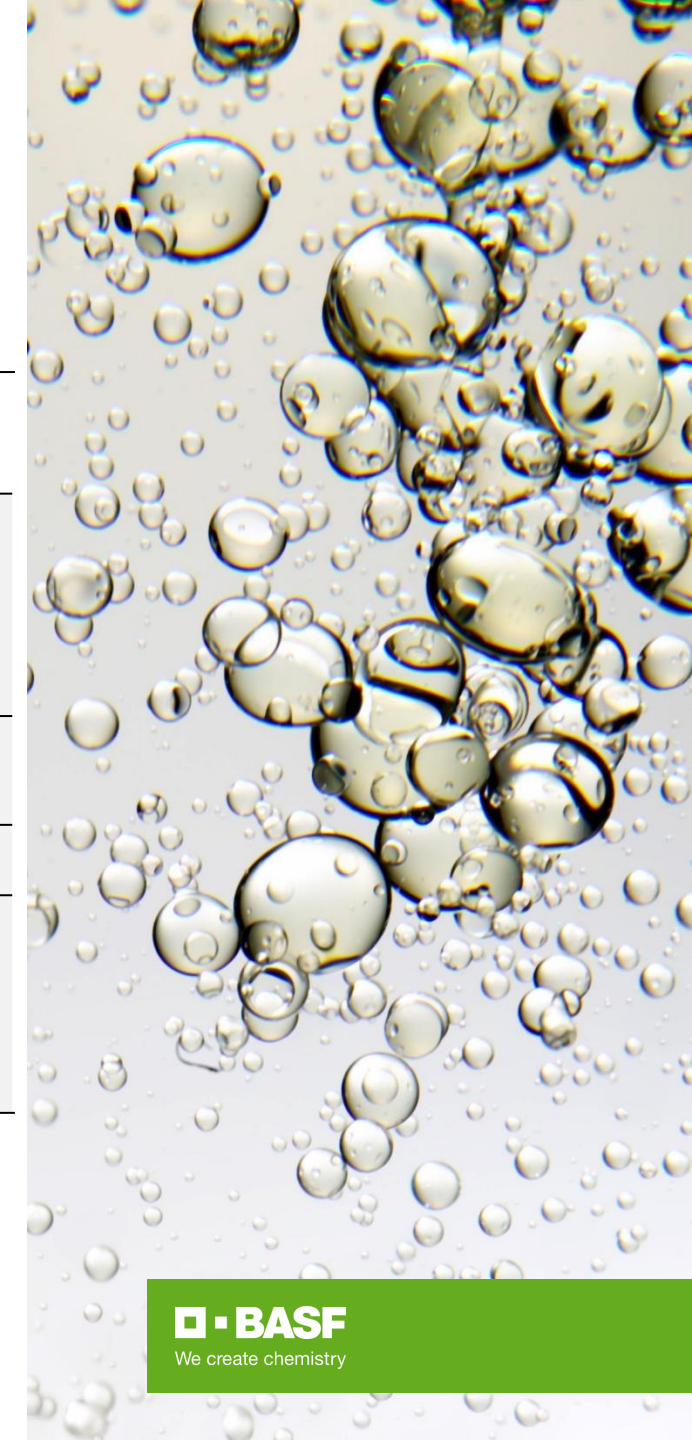
**Enhancing effects** =  $\frac{\text{penetration with surfactant}}{\text{penetration without surfactant}}$

Solvent / base oil	Enhancing Effect
Agnique® AE 829	6.1
Agnique® AMD 10	7.8
Agnique® AE 3-2 EH	7.1
Sorbitan monolaurate, 20 EO	2.5

# Additives for OD formulation

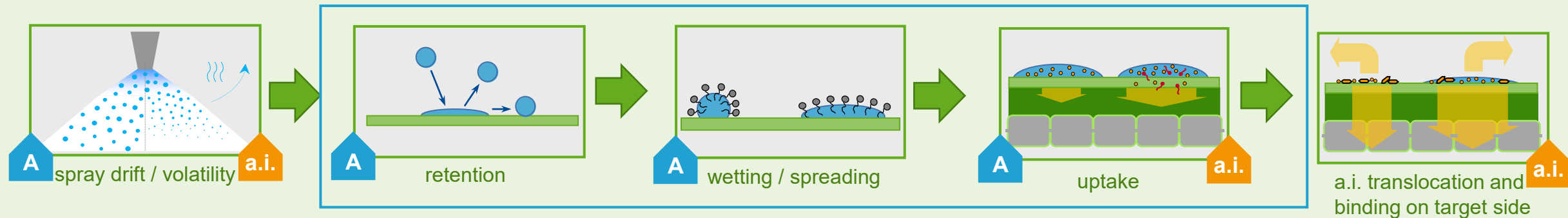
## Non-ionic and ionic emulsifiers

Product	Sustainability profile	Chemistry	Function
Agnique® CSO-20	  	Castor oil ethoxylates	Efficient general-purpose non-ionic emulsifier. Can be combined with CaDDBS
Agnique® CSO-25			
Agnique® CSO-30	 		
Agnique® SPO 40	   	Sorbitol hexaoleate ethoxylate (40 EO)	Good general-purpose label-free emulsifier
Plurafac® LF 403		Oxo alcohol alkoxyate	Emulsifier, penetration agent
Agnique® ABS 70 C		Alkyl benzene sulfonate 70 % in iso-butanol	Anionic emulsifier, typically combined with castor oil ethoxylates
Agnique® ABS 58C-EH		Alkyl benzene sulfonate 58% or 60% in 2-ethylhexanol	
Agnique® ABS 60C-EH			



# Delivery chain

## Adjuvants can not substitute a.i.s or change their mode of action



**A** step affected by adjuvants

**a.i.** step affected by phys.-chem. properties of a.i.

# Adjuvants for OD formulations to further boost a.i. performance

## Recommendations by mode of action

Product	Sustain. profile	Chemistry	Retention	Spreading	Uptake
Agnique® SBO 10		Soybean oil ethoxylate			✓
Agnique® FOH 9 OC-3		Oleyl alcohol ethoxylate			✓
Dehypon® 2574		Fatty alcohol alkoxyate	✓	✓	✓
Dehypon® LS 54		Fatty alcohol alkoxyate	✓	✓	✓
Lutensol® TO 6		Oxo alcohol ethoxylate	✓		
Lutensol® TO 8		Oxo alcohol ethoxylate	✓	✓	✓
Lutensol® TO 10		Oxo alcohol ethoxylate	✓		
Lutensol® XP 30		Guerbet alcohol ethoxylate	✓		
Lutensol® XP 90		Guerbet alcohol ethoxylate	✓		
Lutensol® XL 50		Guerbet alcohol alkoxyate	✓	✓	✓
Lutensol® ON 50		C <sub>10</sub> oxo alcohol ethoxylate		✓	
Plurafac® LF 224		Oxo alcohol alkoxyate		✓	
Plurafac® LF 300		Oxo alcohol alkoxyate		✓	
Plurafac® LF 431		Guerbet alcohol alkoxyate			
Plurafac® LF 901		Guerbet alcohol alkoxyate	✓		
Plurafac® LF 1300		Fatty alcohol alkoxyate			✓
Pluronic® PE 6100		EO-PO-EO block copolymer			

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# OD Formulation:

## FAO required CIPAC tests

Property	Comment	CIPAC Method
Acidity and/or Alkalinity or pH range	In de-ionized water	MT 191 or 75.3
Pourability	Determine maximum residue	MT 148.1
Dispersion stability	Record cream, free oil, sediment, and complete redispersion	MT 180
Wet sieve test	On a $\mu\text{m}$ test sieve	MT 185
Particle size distribution	Share of particles in a certain range	MT 187
Persistent foam	Foam after 1 minute	MT 47.2
Stability at 0°C	Check for crystallisation, separation	MT 39.3
Stability at elevated temperature	14 d at 54 °C, or 28 d at 50 °C, or 8 weeks at 40 °C. Recheck for by-products, acidity/alkalinity, emulsification	MT 46.3
Storage conditions	Freeze/thaw cycles (-10/40 °C), 54°C and RT for 14 d each	

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# Agnique® AE 829

## Emulsification

**Agnique® AE 829** with emulsifier package (Agnique® ABS 70 C + Agnique® CSO-20 + Plurafac® LF 403) shows **instantaneous blooming** on addition to water and **excellent long-term stability (stable after 24 h)**

Instantaneous  
blooming



Emulsion stability  
after 10 inversions (initial)



after 30 minutes



after 4 h



after 24h; ~ 0.5ml  
separation at the top



# Nicosulfuron OD 40 based on Agnique® AE 829

## Test formulation: ODF-S01

Component	Function	% w/v
Nicosulfuron tech. (97.5%)	Active	4.1
Agnique® ABS 70 C	Anionic emulsifier	6.0
Agnique® CSO-20	Non-ionic emulsifier	3.0
Plurafac® LF 403	Non-ionic emulsifier	3.0
Aerosil® R 974	Evonik's rheology agent	4.0
Agnique® AE 829	Base oil	79.9

### Preparation guidance:

Mix Agnique® AE 829 with emulsifiers, homogenize, add Nicosulfuron and Aerosil® 974, pre-disperse with a high-speed mixer/disperser. Grind the suspension with a bead mill to reduce particle size, pH of a 1% aq. solution is 4 – 5.

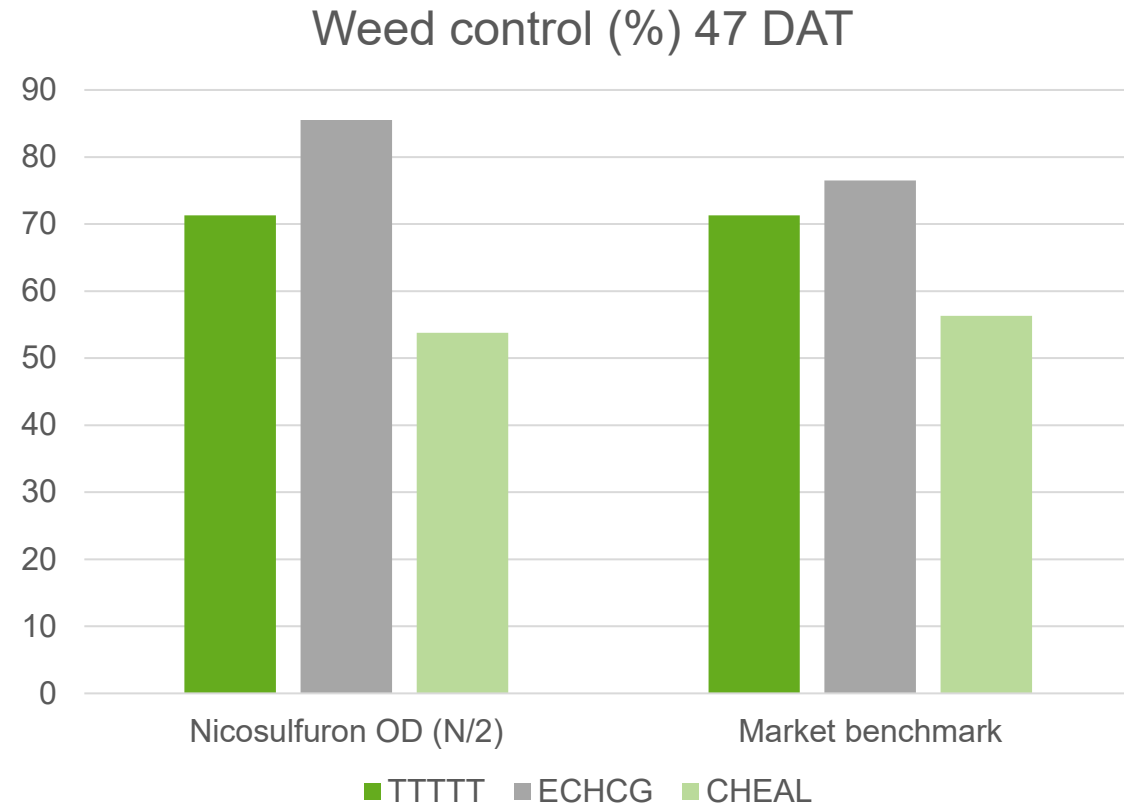




# Nicosulfuron OD 40 (ODF-S01)

## Field trial shows same efficacy as benchmark

Details	Comments
Location	Lake Constance, Germany
GEP	Yes
Crop stage at first application	Corn (var. Sunmark), BBCH 17
Replicates	4
Spray volume	150 L/ha
Nozzles	ARINDL 110-02
Benchmark product	Market benchmark OD 40
Full rate	1 L/ha = N



All treatments were fully selective

# Nicosulfuron OD 40 (ODF-S01)

## Summary

- Stable OD with spontaneous emulsification
  - ▶ Minor increase in viscosity during storage
- Particle size (a.i.) distribution after aqueous dilution cannot be determined by laser diffraction due to interference by larger oil emulsion droplets
  - ▶ However, microscope analysis indicates a homogeneous particle distribution of 1 - 5  $\mu\text{m}$
- Agnique® AE 829 can be used as base oil for a stable OD formulation
  - ▶ Limited degradation of Nicosulfuron meets regulatory requirements
- Biological efficacy meets market benchmark in field trial



# Nicosulfuron OD 240 (ODF-S02) based on MSO

## For EP countries only

Component	Function	% w/w
Nicosulfuron tech. 97.5%	Active	24.6
Agnique® ABS 70 C	Anionic emulsifier	4.0
Agnique® FOH 9 OC-5	Non-ionic emulsifier & penetrant	6.0
Pluronic® A 6000 PE	Oil dispersant	5.0
Aerosil® 200	Evonik's rheology agent	4.0
Agnique® BL 3095	Emulsified base oil	56.4

### Preparation guidance:

Mix Agnique® BL 3095 with emulsifiers, homogenize, add Nicosulfuron and Aerosil® 200, pre-disperse with a high-speed mixer/disperser. Grind the suspension with a bead mill to reduce particle size. A 1% aq. solution in deionized water shows a pH of 4.0 – 4.5.



# Nicosulfuron OD 240 (ODF-S02)

## Properties

- White liquid with viscosity (Brookfield LVT, Sp 2) of
  - ▶ 374 mPa·s at 10 rpm, 226 mPa·s at 30 rpm, 146 mPa·s at 100 rpm
- Excellent emulsification
- Stability after 8 weeks at 35 °C
  - ▶ No sediment/no gelling
  - ▶ 9% separation
- Degradation of Nicosulfuron
  - ▶ Benchmark at -18 °C                    24.4% w/w
  - ▶ After 14 d at 54 °C                    24.4% w/w
  - ▶ Degradation of the active:        0%

## Summary

- Using standard ingredients, stable OD based on methylated seed oil obtained
- All components comply with REACh



# OD Formulation: Trouble shooting

Problem	Solution suggestion
Sedimentation	Increase the level of rheology modifier or change it. Always check viscosity and spontaneity of aq. dispersion.
Gelling	Adjust or change of emulsifier system.
Poor emulsion	If an increased emulsifier concentration does not help, change the emulsifier system.
Solid particles aggregate and adhere to the inner glass surface	Upon preparation of the spray solution, flocculation can occur. Optimization of emulsifiers is preferred or try to add an aq. dispersant.

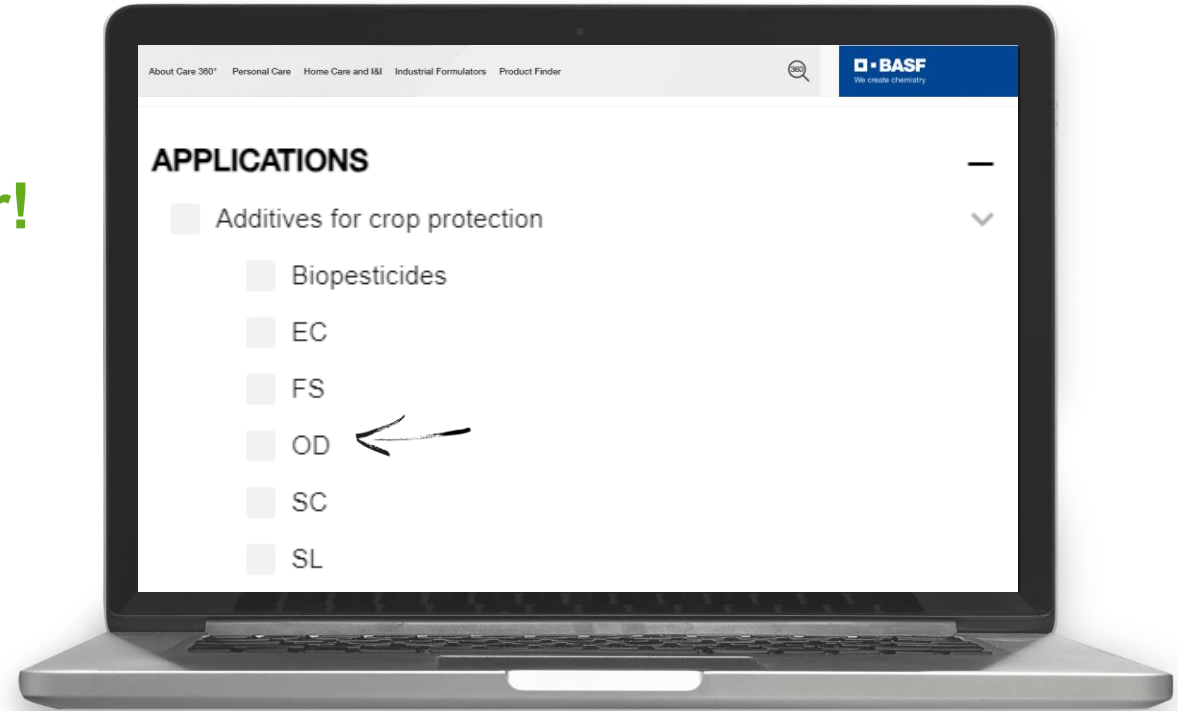
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