Advanced Formulation Technology and its benefits for Clomazone containing herbicides

Fortschrittliche Formulierungstechnologie und ihr Nutzen für Clomazone-haltige Herbizide

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Abstract

Clomazone is an important compound for effective weed control in winter oilseed rape and spring crops as potatoes and vegetables. Both when applied solo and as a complementary partner to other active ingredients, clomazone offers good and reliable control on a range of key weed species and crop safety. Its unique mode of action brings valuable contribution to anti-resistance weed management strategies. Clomazone effects on susceptible weed species are the typical bleaching symptoms. FMC developed and patented a unique CS microencapsulation technology for clomazone formulations. This technology as used in Centium 36 CS maintains efficacy and crop safety and reduces the risk of potential damage to non-target plants. In addition FMC introduces two novel formulation platforms, Synchronized Technology (SYNC^{TEC}) and Dual Active Matrix Technology (DAM^{TEC}). Synchronized Technology (SYNC^{TEC}) means co-microencapsulation of multiple active ingredients and synchronized delivery to the target after application, hence making the different herbicides available at their optimal timing. Dual Active Matrix Technology (DAM^{TEC}) combines a microencapsulated active ingredient with a second active ingredient in granular form. Both proprietary technologies are specifically designed for co-formulated products and preserve the unique properties of the different active ingredients and all benefits from the CS microencapsulation system including control of volatility and high efficacy performance.

Keywords: Clomazone, dual active matrix, microencapsulation, performance, secondary drift control, synchronized delivery, technology platforms

Zusammenfassung

Clomazone ist ein wichtiger Wirkstoff für eine effektive Unkrautbekämpfung in Winterraps und Sommerkulturen wie Kartoffel und Feldgemüsearten. Sowohl bei alleiniger Anwendung wie auch als Partner anderer Wirkstoffe bietet Clomazone eine gute und sichere Bekämpfung wichtiger Unkrautarten bei guter Kulturverträglichkeit. Der besondere Wirkmechanismus liefert einen wesentlichen Beitrag zu Anti-Resistenzstrategien in der Unkrautbekämpfung. Der Effekt von Clomazone auf empfindliche Unkrautarten äußert sich in den typischen Entfärbungssymptomen (*bleaching*) an grünen Pflanzenteilen. Für Formulierungen, die als Wirkstoff Clomazone enthalten, hat FMC hat eine besondere CS Mikroverkapslungstechnologie entwickelt und patentieren lassen. Die Technologie, wie sie bei *Centium 36 CS* zur Anwendung kommt, unterstützt die Wirkung und Kultursicherheit und reduziert das Risiko eines potentiellen Schadens an Nicht-Zielpflanzen. Mit der ‚*Synchronized Technology* (SYN^{TEC})' und der ‚*Dual Active Matrix Technology* (DAM^{TEC})' begibt sich FMC auf zwei neuartige Ebenen in der Formulierungstechnologie. Unter der ‚*Synchronized Technology* (SYN^{TEC})' ist die gemeinsame Mikroverkapselung mehrerer Wirkstoffe und ihre aufeinander abgestimmte Lieferbarkeit nach Applikation zu verstehen, während bei der *Dual Active Matrix Technology* (DAM^{TEC}) eine Mikroverkapselte Aktivsubstanz mit einem in Granulatform vorliegenden Wirkstoff kombiniert werden. Beide proprietären Technologien sind speziell für co-formulierte Produkte ausgelegt, um sowohl die speziellen Wirkeigenschaften der Einzelkomponenten als auch alle Vorteile der CS-Verkapselung zu erhalten wie etwa die induzierte Abdrift-Reduktion und die hohe Wirkleistung.

Stichwörter: Aufeinander abgestimmte Vorrätigkeit, Clomazone, *Dual Active Matrix* (duale active Matrix), induzierte Abdrift-Reduktion, Leistung, Mikroverkapselung, Technologie-Ebenen

Introduction

Since many years the systemic and selective herbicide clomazone is used to control broad-leaved and grass weeds in a range of important agricultural crops such as oilseed rape, potato and

vegetables. Applied in pre-emergence of the crops with its unique mode of action (HRAC classification F4 – Inhibition of DOXP Synthase; FERHATOGLU and BARRETT, 2005) Clomazone is uptaken by the roots and shoots of the emerging weeds and subsequently acropetal translocation it interferes with the production and protection of the photosynthetic pigments and this results in the typical whitening or bleaching symptoms of the target weeds before they die off. Clomazone is an important corner stone in the farmers weed control programs due to its efficacy on difficult to control weeds such as cruciferous and *Polygonum* species. Hence it is an interesting partner for other pre-emergence herbicide compounds to provide broad spectrum weed control.

The active ingredient clomazone was introduced in Europe in 1991 as EC (emulsifiable concentrate) and WP (wettable powder) formulated commercial products. When plant protections products (PPP's) are sprayed, drift to non-target sites is recognized as one of some potential undesirable side effects. Drift originates from primary (wind) drift and/or secondary drift (volatilisation). For clomazone containing products the symptoms are characterized by bleaching of susceptible non-target plants such as *Stellaria media*. In most situations the reasons for drift are applicator related (FISHEL and FERREL, 2013). In 2001 Centium 36 CS (registration number 004798-00) obtains its first registration in Germany. Centium 36 CS is a proprietary microencapsulation technology that is specifically designed to reduce the secondary off-target movement potential of the active ingredient Clomazone. Various levels of evaluation; including but not limited to laboratory, greenhouse and field environment; are imbedded within the microencapsulation development and quality validation process to confirm the performance standards for both efficacy, target crop safety and secondary drift control.

Two upcoming microencapsulation based technologies have been developed to provide multiple active ingredient broad spectrum herbicide mixtures that are attractive for practical use, whereby clomazone is one of the ingredients. Essential compared to common tank mixtures is that due to the unique combinations and the optimal balanced amount of active ingredients, the individual formulated products lead to very high efficacy against a wide range of weeds, have very good selectivity in targeted registered crops and provide desired secondary drift control.

SYNC^{TEC} or Synchronized Delivery to Target technology is a state-of-the art proprietary technology in the form of a co-microencapsulation (CS) that delivers two or more active ingredients at the optimal timing. This water-based innovative formulation technology contains specifically designed amounts of the composite active ingredients and secures the herbicidal properties for each of them. The effect of the co-microencapsulation is characterized by a synchronized availability of the individual active ingredients after spraying to the soil surface in order to bring the expected high level performance for weed control and target crop safety. Complementary the SYNC^{TEC} platform provides the desired reduction of secondary off-target movement potential for clomazone active ingredient that meets the Centium 36 CS formulation development standard.

DAM^{TEC} or Dual Active Matrix technology represents a breakthrough patented formulation technology that combines clomazone in a micro-encapsulated form with a second active ingredient in a convenient to use granular formulation. The porous granules represent an optimized delivery system of the considered active ingredients until the moment of application under field conditions. The result in the field is the expected immediate and residual weed control with the excellent control of secondary off-target movement for clomazone.

The desired features for the formulation candidates are tested and evaluated in under different environmental conditions that are representative for the post-sowing pre-emergence weed control in both spring and autumn field conditions.

Material and Methods

Field experiments to demonstrate importance of microencapsulation quality

Based on the EPPO guideline PP 1/256(1), Ganzelmeier investigations on spray drift (GANZELMEIER *et al.*, 1995) and according to the revised tables of new basic drift values (RAUTMANN *et al.*, 2001) one

small plot replicated field trial was executed in spring 2013 in Vregny, France, to evaluate the potential of non-target plant damage following spray or wind drift. The experiment was carried out under a randomized complete block (RCB) design with 2 replicates and individual plot size of 6m². Centium 36 CS based on proprietary microencapsulation technology, a non-FMC clomazone microencapsulated formulation and a non-microencapsulated clomazone formulation were directly sprayed on a sugar beet crop (cultivar: Julietta) at growth stage BBCH12 at dose rates equivalent to the drift values calculated according to Ganzelmeier and related to distances of 1, 3, 5, 10 and 15 metres on the basis of the spring label recommended dose rate (i.e. 1N or 90 g clomazone/ha) of Centium 36 CS. Additionally, the trial comprised an extra treatment for the 2N dose related to the distance of 1 metre. Within an interval from 2 days until 1 month after application several assessments on each experimental plot were carried out at regular intervals to determine the extend of phytotoxic responses on the sugar beet crop.

Furthermore in 2012 and 2013, both under spring and autumn agroclimatic conditions additional unreplicated experiments were conducted according to the ‚field barrel'test methodology developed by FMC. Centium 36 CS and a non-FMC clomazone microencapsulated formulation were evaluated for their potential to reduce the off-target movement of Clomazone under German field conditions. The reference non-microencapsulated Clomazone formulation allows for relative comparison. All test formulations were applied to the soil at equivalent active ingredient rates on an area of 4 to 20m² representative for post-sowing pre-emergence weed control applications in spring grown crops and also in winter oilseed rape. Applications were made in alignment with farmer good agricultural practice recommendations and current German national label mitigation measures for clomazone containing herbicides. In addition wind screens were placed to exclude any risk for wind drift. Pots containing evenly germinated *Stellaria media* plants were placed at regular intervals of 0.5m from the treated area in the shape of a radius according to the different wind spokes. *Stellaria media* as indicator plant species were used due to its high susceptibility to low amounts of clomazone. At regular time intervals from application onwards assessments of the *Stellaria media* bleaching were made for each pot in each direction at any distance.

Field experiments to confirm efficacy, crop safety and secondary drift control with SYNC^{TEC} and DAM^{TEC}

A global multi-year data set from 2009 to 2012 with trials spread over Europe and conducted in maritime, mediterranean, north-eastern and south-eastern EPPO climatic zone is used to evaluate both state-of-the-art technologies SYNC^{TEC} and DAM^{TEC} for their efficacy and target crop safety under different representative field conditions. Replicated small plot field trials were laid out in randomized complete block design with 3-4 replicates per treatment and were executed according to the relevant EPPO guidelines (PP 1/135, 1/152, 1/049, 1/050, 1/051, 1/051, 1/053, 1/091 and 1/099). Experiments were located in fields of different commercial grown target crops with natural weed infestations covering winter oilseed rape, potato, peas, field beans, carrots, maize and soybeans and located in representative growing areas. Each time a single application was carried out in pre-emergence of crop and weeds. To assess efficacy and crop safety for DAM^{TEC} 94 trials; 22 in 2009, 15 in 2010, 30 in 2011, 27 in 2012; and for SYNC^{TEC} 225 trials whereby 51 in 2009, 37 in 2010, 61 in 2011 and 76 in 2012; were included in the analysis. Weed control and crop safety assessments were rated in percentage (scale 0-100) compared to the untreated control plots. The experimental products were applied at the 1N target rates. Corresponding rate-for-rate tank mixtures with Centium 36 CS were included in the treatment list as reference products.

To test the potential of the SYNC^{TEC} and DAM^{TEC} technologies to reduce the secondary off-target movement of clomazone, in autumn 2012 two unreplicated field experiments following the earlier described ‚field barrel' test methodology were organized in Germany under conditions representative for post-sowing pre-emergence herbicide applications in winter oilseed rape.

Results

Field experiments to demonstrate importance of microencapsulation quality

The simulation of spray drift (*Ganzelmeier* drift values) after a pre-emergence application with respectively Centium 36 CS, a non-FMC microencapsulated CS formulation and non-microencapsulated reference product, respectively, at spring label recommended rate (i.e. 90 g clomazone/Ha) demonstrate differences in undesired phytotoxic effect on adjacent growing non-target plant species generated by equivalent rates of Clomazone active ingredient at identical distances from site of application (Fig. 1).

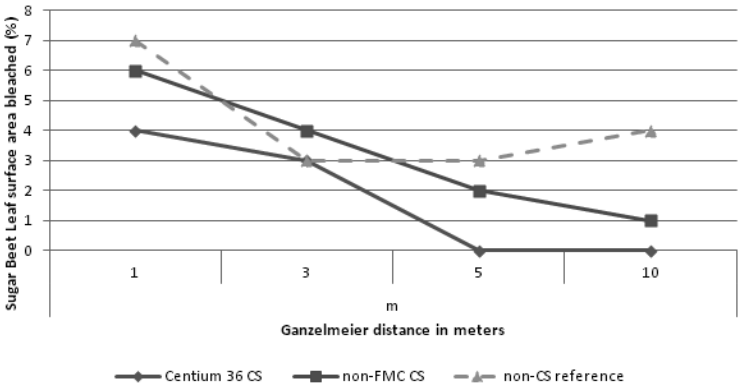

Fig. 1 Primary drift damage sugar beet – percentage (%) leaf bleaching with distance from treated area (10 days after application, Sugar beet (Bayer code: BEAVA, BBCH 14-16).

Abb. 1 *Primärschaden durch Abdrift an Zuckerrübe – Prozent (%) Blattentfärbung mit Abstand zur behandelten Fläche (10 Tage nach Applikation, Zuckerrübe (Bayer code: BEAVA, BBCH 14-16).*

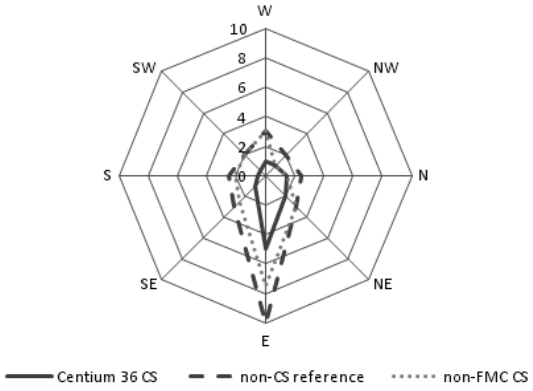

Fig. 2 Area (0-25 m) of clomazone leaf bleaching effect on Stellaria media indicator plant species between Centium 36 CS, non-FMC CS-formulation and non-CS standard (31 days after application).

Abb. 2 *Vergleich der durch Clomazone hervorgerufenen Bleaching-Bereiche (0-25 m) an der Indikator-Pflanzenart Stellaria media nach Anwendung von Centium 36 CS vs. nicht-FMC-CS-Formulierung vs. nicht-CS-Standard (31 Tage nach Applikation).*

In Figure 2 the maximum distance and surface area that can potentially be impacted by secondary drift or volatilization after a pre-emergence autumn application (Hönow-Brandenburg, 2012) highlights significant relative differences between tested formulations. Results are based on *Stellaria media* indicator species leaf bleaching assessed at regular distances from site of application up to 25 meters from the application area in the different wind directions. Compared to the non-microencapsulated reference product Centium 36 CS reduces the size of the impacted area with approximately 60% while for the non-FMC microencapsulation this is only 19% smaller. Hence under both spring and autumn agroclimatic conditions the average reduction of size of potential impacted area is 66% for Centium 36 CS and 35% for the non-FMC microencapsulated formulation.

Field experiments to confirm efficacy, crop safety and secondary drift control with SYNC^{TEC} and DAM^{TEC}

The efficacy of four different SYNC^{TEC} formulations, each characterized by a unique and balanced combination of two or more active ingredients including one is clomazone, that are developed to bring broad range weed control in a variation of target crops after a post-sowing pre-emergence application at the 1N target field rate, confirms to preserve the desirable herbicidal activity of the individual compounds (Tab. 1).

Tab. 1 Efficacy (%) SYNC^{TEC} versus common tank mixture at different intervals after application (DAA – days after application) when applied in post-sowing pre-emergence and variability (2009-2012).

***Tab. 1** Vergleich der Wirksamkeit (%) von SYNC^{TEC} vs. herkömmlicher Tankmischung zu unterschiedlichen Terminen (DAA – Tage nach der Applikation) nach Vorauflaufanwendung einschließlich der Schwankungsbreiten (2009-2012).*

	SYNC^{TEC}					**Tank mixture**				
DAA	28-42	56-84	84-112	112-168	> 168	28-42	56-84	84-112	112-168	> 168
Mean	80.16	83.13	73.3	78.66	87.38	81.1	83.79	73.21	77.86	90.96
Median	91	93.75	81.25	86.38	97	88.75	95	80.99	85.88	99.25
Stdev	25.24	24.09	28.33	23.72	22.8	23.72	23.24	27.35	23.65	18.96
Q25	72.5	76.88	59.13	66.84	85.36	75.63	77.5	58.22	65.04	92.79
Q75	98.25	99.5	96.75	97.5	100	98.25	99.75	96.38	97.79	100
N (trials)	*118*	*123*	*60*	*31*	*53*	*118*	*123*	*60*	*31*	*53*
N (weeds)	*574*	*619*	*291*	*120*	*247*	*574*	*619*	*291*	*120*	*247*
Student T-tests (p-value)	0.51	*0.62*	*0.85*	*0.79*	*0.06*					
Result comparison	=	=	=	=	=					

For the evaluation all findings are cumulated over all trials and assessed weed species (79 individual species) during defined assessment intervals that reflect short-, medium- and long-term control, in order to compare the total mean efficacy levels and dispersion of the SYNC^{TEC} technology with its corresponding common tank mixture including Centium 36 CS.
In Table 2 a comparison of the crop tolerance is shown for SYNC^{TEC} formulations and the common tank mixture. It is evident that after a single pre-emergence application at the 1N target rate the leaf bleaching as main effect is restricted to low levels, i.e. maximum average values of below 4%, transient (FMC AGRICULTURAL PRODUCTS GROUP, 2000) and within limits of grower's acceptance.

Comparable findings result for the SYN^{TEC} engineered formulations and the common tank mixtures at rate-for-rate applications.

The efficacy generated during the development process of two different DAM^{TEC} formulations, each characterized by a unique and balanced combination of two active ingredients hence one is clomazone, to bring broad range weed control in a selection of target crops after a post-sowing pre-emergence application at the 1N target field rate, confirms to preserve the desirable herbicidal activity of the individual compounds (Tab. 3). For the evaluation all findings are cumulated over all trials and assessed weed species (53 individual species) during defined assessment intervals that reflect short-, medium- and long-term control, in order to compare the total mean efficacy levels and dispersion of the DAM^{TEC} technology with its corresponding common tank mixture.

Tab. 2 Crop safety (% bleaching) SYNC^{TEC} versus common tank mixture at different intervals after application (DAA – Days After Application) when applied in post-sowing pre-emergence and variability (2009-2012.)

***Tab. 2** Vergleich der Kulturverträglichkeit (% Bleaching) von SYNC^{TEC} vs. herkömmlicher Tankmischung zu unterschiedlichen Terminen (DAA – Tage nach der Applikation) nach Vorauflaufanwendung einschließlich der Schwankungsbreiten (2009-2012).*

	SYNC^{TEC}				**Tank mixture**			
DAA	14-22	28-42	55-72	84-112	14-22	28-42	55-72	84-112
Mean	3.61	1.52	0.68	0.08	3.26	1.9	0.76	0.08
Median	0	0	0	0	0	0	0	0
Stdev	10.0	4.2	2.1	0.53	9.1	5.8	2.4	0.53
Maximum	75.0	28.75	15	3.75	65	40	13.75	3.75
Q25	0	0	0	0	0	0	0	0
Q75	2.6	0.67	0	0	2.8	0.75	0	0
N (trials)	*112*	*175*	*94*	*49*	*112*	*175*	*94*	*49*
Student T-test (p-value)	*0.67*	*0.39*	*0.7*	*1*				
Result comparison	=	=	=	=				

Visual crop tolerance is shown for DAM^{TEC} formulations and the common tank mixture after a single pre-emergence application at the 1N target rate is evaluated for the leaf bleaching as main effect (Tab. 4). The overall crop reaction indicates high crop safety with maximum average values below 2% and typically any occurring symptoms are transient, this means disappearing shortly afterwards without any impact on crop development or yield.

A statistical test has been run to compare the SYNC^{TEC} and DAM^{TEC} technology with the corresponding tank mixture with Centium 36 CS, using the statistical software RStudio (Integrated development environment for R, version 097.247). To compare the means at each interval considered, the Student's T-test has been applied (95% confidence interval), given that more than 30 data points were used to build the means. Each time the results of the tests are given at the end of the table comparisons via the p-value and via the direction of the comparison ('=' means that averages are not statistically different, '<' means the average is inferior to the Tank mix, '>' means the average is superior to the Tank mix).

Tab. 3 Efficacy (%) DAM^{TEC} versus common tank mixture at different intervals after application (DAA – days after application) when applied in post-sowing pre-emergence and variability (2009-2012).

***Tab. 3** Vergleich der Wirksamkeit (%) von DAM^{TEC} vs. herkömmlicher Tankmischung zu unterschiedlichen Terminen (DAA – Tage nach der Applikation) nach Vorauflaufanwendung einschließlich der Schwankungsbreiten (2009-2012).*

	DAM^{TEC}					**Tank mixture**				
DAA	**28-42**	**56-84**	**84-112**	**112-168**	**> 168**	**28-42**	**56-84**	**84-112**	**112-168**	**> 168**
Mean	87.72	90.89	77.54	75.25	100	86.58	89.03	74.21	75.87	100
Median	98.25	98	90	90		97.5	98.75	90.63	90	
Stdev	21.45	15.73	31.85	34.17		22.54	19.36	33.74	34.50	
Q25	84.45	87.5	73.6	65.07		83.61	86.88	55.63	54.16	
Q75	100	100	98.97	99		100	100	99	99.88	
N (trials)	*60*	*51*	*24*	*10*	*1*	*60*	*51*	*24*	*10*	*1*
N (weeds)	*295*	*303*	*118*	*55*	*6*	*295*	*303*	*118*	*55*	*6*
Student T-test (p-value)	*0.46*	*0.14*	*0.21*	*0.92*	*NA*					
Result comparison	=	=	=	=						

Tab. 4 Crop safety (% bleaching) DAM^{TEC} versus common tank mixture at different intervals after application (DAA – days after application) when applied in post-sowing pre-emergence and variability (2009-2012).

***Tab. 4** Vergleich der Kulturverträglichkeit (% Bleaching) von DAM^{TEC} vs. herkömmlicher Tankmischung zu unterschiedlichen Terminen (DAA – Tage nach der Applikation) nach Vorauflaufanwendung einschließlich der Schwankungsbreiten (2009-2012).*

	DAM^{TEC}				**Tank mixture**			
DAA	14-22	28-42	55-72	84-112	14-22	28-42	55-72	84-112
Mean	1.99	1.43	0.23	0	1.65	1.06	0.21	0
Median	0	0	0	0	0	0	0	0
Stdev	4.74	4.37	0.85	0	4.36	3.60	0.70	0
Maximum	21.25	25	5	0	18.75	25	3.33	0
Q25	0	0	0	0	0	0	0	0
Q75	0.25	0	0	0	0	0	0	0
N (trials)	*34*	*61*	*40*	*20*	*34*	*61*	*40*	*20*
Student T-test (p-value)	*0.74*	*0.58*	*0.94*	*NA*				
Result comparison	=	=	=					

Discussion

Current situation for plant protection products and active ingredients is challenging. On the one hand they are expected to solve the agronomic problems by long term activity with a minimum of

applications; on the other hand their potential impact on the environment is viewed with suspicion. Undesired damage to non-target areas and plant species is amongst them. Under these conditions formulation technology and its optimization is increasingly important. Clomazone is used since many years to control broad-leaved and grass weeds in a range of important agricultural crops such as oilseed rape, potato and vegetables and hence acts as an important pillar and in farmers weed control strategies. Drift to non-target sites is recognized as one of some potential undesirable side effects. Drift originates from primary (wind) drift and/or secondary drift (volatilisation). For Clomazone containing products the symptoms are characterized by bleaching of susceptible non-target plants. While valuable mitigation measures such as but not limited to drift reducing nozzles, appropriate driving speed and minimum spray water volumes; are available to the farmer to limit the potential of primary drift; to reduce the risk for secondary drift inherently related to the physical/chemical properties of the active ingredient, only formulation technology provides relief. Microencapsulated formulation technology (CS) can reduce the secondary off-target movement potential of the active ingredient clomazone. Centium 36 CS is a proprietary microencapsulation technology that is specifically designed to provide reliable control of clomazone off-target movement. Various evaluation levels; including but not limited to laboratory, greenhouse and field environment; are imbedded within the microencapsulation development and quality validation process to confirm the internal set performance standards. An individual field trial set-up in Vregny in France investigated the impact on crop safety after a direct application of different spray drift rates with Centium 36 CS, a non-FMC clomazone microencapsulated formulation and a non-microencapsulated clomazone formulation on a sugar beet crop at growth stage BBCH12, representing the non-target adjacent crop species. Spray drift rates were calculated according to GANZELMEIER *et al.* (1995). From the assessments it became apparent that equivalent rates of active ingredient result in different impact on the non-target crop, with Centium 36 CS provoking the lowest levels of crop reaction by means of leaf bleaching and most rapidly decreasing with distance from site of application. To assess the potential of SYNC^{TEC} and DAM^{TEC} technologies to reduce volatilization two individual field trials, located in Mulsum-Niedersachsen and Hönow-Brandenburg, were laid down in 2012 under conditions representative for pre-emergence weed control strategies in winter oilseed rape in Germany. Compared to a non-microencapsulated formulation the maximum distance and surface area that can potentially be impacted by secondary drift is reduced through FMC proprietary microencapsulated formulations in average by 63% for the Centium 36 CS, 70% for the SYNC^{TEC} and 88% for DAM^{TEC} developments. Hence the risk and size of potential impacted area is significantly reduced. These field trials demonstrate significant better performance for FMC proprietary microencapsulation technology what can be translated into reduced risk of undesired damage to non-target plant species following normal farmers practice applications regardless the type of drift. By consequence the formulation development process encompasses now more than ever the balanced combination of optimized biological efficacy and crop safety with safe and convenient-to-use formulations.

From 2009 to 2012 for SYNC^{TEC} and DAM^{TEC}, respectively, 225 and 94 individual field trials including 79 and 53 different weed species were laid down spread across maritime, mediterranean, north-eastern and south-eastern EPPO climatic zones in Europe to compare their efficacy for weed control and crop safety with common tank mixtures with Centium 36 CS when applied at the 1N target rate. A single pre-emergence application in a range of target crops with natural weed infestations covering winter oilseed rape, potato, peas, field beans, carrots, maize and soybeans, did confirm the statistical equivalence, by Student's T-test mean comparisons, how the technology retains the benefit of the activity of the active ingredients without any compromise on crop safety.

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