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Technical Note

PHYSICOCHEMICAL COMPATIBILITY OF AGROCHEMICAL MIXTURES IN SPRAY TANKS FOR PADDY FIELD RICE CROPS

Compatibilidade Físico-Química de Misturas de Agrotóxicos em Tanque de Pulverização para Cultura do Arroz Irrigado

RAKES, M.[1]*
GRÜTZMACHER, A.D.[1]
PAZINI, J.B.[1]
PASINI, R.A.[1]
SCHAEDLER, C.E.[2]

ABSTRACT - The use of pesticide mixtures constitutes a relatively common practice in rice crops. Thus, the aim of this work was to evaluate the physicochemical interaction among different pesticide tank mixes for use in paddy field rice. The study has followed technical standards specified in ABNT [Associação Brasileira de Normas Técnicas (Brazilian National Standards Organization)] NBR [Norma Brasileira Regulamentadora (Brazilian Regulatory Standard)] NBR 13875:2014 for the assessment of physicochemical compatibility by means of a dynamic technique. Treatments consisted of mixtures of 12 pesticides, which constituted 16 treatments, six of which are composed by mixing herbicides, six by mixing herbicide and insecticide, one by mixing fungicides, and three by mixing fungicide and insecticide. Tank mixtures among herbicides Clincher® + Ricer®, Clincher® + Kifix®, Clincher® + Imazethapyr Plus Nortox®, Clincher® + Ricer® + Kifix®, Clincher® + Ricer® + Sirius® 250 CS, Imazethapyr Plus Nortox® + Basagran® 600, between herbicides and insecticides Clincher® + Ricer® + Arrivo® 200 EC, Clincher® + Kifix® + Arrivo® 200 EC, Clincher® + Imazethapyr Plus Nortox® + Arrivo® 200 EC, Clincher® + Ricer® + Kifix® + Arrivo® 200 EC, Clincher® + Ricer® + Sirius® 250 CS + Arrivo® 200 EC, Imazethapyr Plus Nortox® + Basagran® 600 + Arrivo® 200 EC, among fungicides Alterne® + Bim® 750 BR + Priori® 250 CS, and between fungicides and insecticides Bim® 750 BR + Actara® 250 WG, Alterne® + Bim® 750 BR + Priori® 250 CS + Actara® 250 WG, and Alterne® + Bim® 750 BR + Priori® 250 CS + Talisman® did not present any physicochemical change in the spray mix and are therefore compatible to be used in mixtures in the spray tank in plant treatments in rice crops.

Keywords: *Oryza sativa,* herbicide, insecticide, fungicide, incompatibility

RESUMO - O uso de agrotóxicos em mistura no tanque de pulverização constitui prática comum na orizicultura. O objetivo desse trabalho foi avaliar a compatibilidade físico-química de misturas de agrotóxicos em tanque de pulverização para a cultura do arroz irrigado. O estudo seguiu os padrões técnicos especificados na norma da Associação Brasileira de Normas Técnicas – NBR 13875:2014 para avaliação de compatibilidade físico-química por meio da técnica dinâmica. Os tratamentos foram constituídos pelas misturas de 12 agrotóxicos, num total de 16 tratamentos, sendo seis compostos pela mistura de herbicidas; seis pela mistura de herbicida e inseticida; um pela mistura entre fungicidas; e três pela mistura de fungicida e inseticida. As misturas entre herbicidas Clincher®+Ricer®, Clincher®+Kifix®, Clincher®+Imazetapir Plus Nortox®, Clincher®+Ricer®+Kifix®, Clincher®+Ricer®+Sirius® 250 SC, e Imazetapir Plus Nortox®+Basagran® 600; entre herbicida e inseticida Clincher®+Ricer®+Arrivo® 200 EC, Clincher®+Kifix®+Arrivo® 200 EC, Clincher®+Imazetapir Plus

*** Corresponding author:**
<matheusrakes@hotmail.com>

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[1] Universidade Federal de Pelotas (UFPel), Pelotas-RS, Brasil; [2] Universidade Federal do Pampa, (UNIPAMPA), Itaqui-RS, Brasil.

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Nortox®+Arrivo® 200EC, Clincher®+Ricer®+Kifix®+Arrivo® 200 EC, Clincher®+Ricer®+Sirius® 250 SC+Arrivo® 200 EC e Imazetapir Plus Nortox®+Basagran® 600+Arrivo® 200 EC; entre fungicidas Alterne®+Bim® 750 BR+Priori® 250 SC; e entre fungicida e inseticida Bim® 750 BR+Actara® 250 WG, Alterne®+Bim® 750 BR+Priori® 250 SC+Actara® 250 WG e Alterne®+Bim® 750 BR+Priori® 250 SC+Talisman® não apresentaram qualquer alteração físico-química na calda e, portanto, são compatíveis para ser empregadas em tratamentos fitossanitários na cultura do arroz.

Palavras-chave: *Oryza sativa*, herbicida, inseticida, fungicida, incompatibilidade.

INTRODUCTION

Rice is the second most cultivated cereal in the world. Among biotic factors that reduce its productivity are weeds, insects and diseases. In order to reduce the damage caused by these pests, the most widely used method is chemical control with the use of agrochemicals. It is known, however, that under plowing conditions these pests occur simultaneously and chemical control becomes difficult and costly because each of these biological agents needs products of specific classes. Thus, it is necessary to use more than one agrochemical mixed in a spray tank (Ronchi et al., 2002; Petter et al., 2013) so that the set of crop protection problems be controlled at reduced costs.

It is known that herbicide and insecticide applications after emergence of rice are considered frequent practices in paddy field rice paddies in the South of Brazil, aiming the control of weeds and fall armyworm (*Spodoptera frugiperda*) (J. E. Smith, 1797) (Lepidoptera: Noctuidae) in the pre-flood period of crops, which is exactly the period in which the insect is most damaging to the crop (Botton et al., 1998). Also, as a measure of reduction of production costs, tank mixing of fungicides and insecticides is common in paddy field rice crops for the simultaneous control of rice blast fungus (among other common names) (*Magnaporthe oryzae* B. Couch) and of stink bugs such as *Tibraca limbativentris* Stal, 1860 (Hemiptera: Pentatomidae) and *Oebalus* spp. (Hemiptera: Pentatomidae).

Tank mixing is the associations of agrochemicals and the like in the applicator equipment tank just before the application. Currently this practice is not prohibited but it is farmers' responsibility (Petter et al., 2013) and can not be prescribed in an agronomic recipe (Gazziero, 2015). This is in line with Brazilian Ruling 4074/02 (Brazil, 2002), according to which any agrochemical can only be prescribed by a legally qualified professional and the products may be prescribed only in accordance with recommendations of use approved in the package insert.

In a case study, mixtures of agrochemicals in a spray tank are frequently carried out on different agricultural crops in Brazil (Gazziero, 2015). Also, 95% of the mixtures include the use of two to five agrochemicals. In some cases, this number is higher than six agrochemicals. However, this practice may represent serious losses to producers due to the occurrence of incompatibilities among chemical compounds in the spray mix (Ronchi et al., 2002; Castro, 2009; Petter et al., 2013). There are at least two types of incompatibility that can occur in tank mixing, which are: physical incompatibility, which corresponds to the formation of precipitates or granules; chemical incompatibility, which occurs when tank mixing alters the efficiency of the active ingredients blended. Physical incompatibility may lead to clogging of spray nozzles and filters and, consequently, loss of product efficacy and difficulties during application (Gazziero, 2015). On the other hand, cases of chemical incompatibility can generate an effect of increasing toxicity, causing damage to the crop and decreasing productivity potential (Trezzi et al., 2005; Petter et al., 2012).

It should be noted, however, that the incompatibility of plant protection products is primarily physical. Thus, the physical compatibility of the pesticides is only the first of the events that rule the stability of the spray mix and, consequently, the effects on the biological target (Petter et al., 2012). However, it is known that the information about physical compatibility of the spray in a tank mix of different agrochemicals used in rice cultivation and its effects is scarce. Thus,

the objective of this study was to evaluate the physicochemical compatibility of mixtures of agrochemicals in a spray tank for paddy field rice cultivation.

To evaluate the physical compatibility of agrochemicals in the spray tank, 12 agrochemicals (six herbicides, three insecticides and three fungicides) registered at Brazilian federal department Ministry of Agriculture, Livestock, and Supply (Ministério da Agricultura, Pecuária e Abastecimento, abbreviated MAPA) for rice and/or paddy field rice (AGROFIT, 2016), which constituted 16 treatments, six of which were compounded by a herbicide mixture, six by a mixture of herbicide and insecticide, one by a mixture among fungicides, and three by a mixture of fungicide and insecticide (Table 1). The choice of agrochemical mixtures occurred because of the frequent use by farmers, proven by aerial spraying reports from the 2014/15 crop provided by an agricultural airplane company located in the southern region of Brazil, in the state of Rio Grande do Sul.

In the tests, technical standards specified in ABNT [Associação Brasileira de Normas Técnicas (Brazilian National Standards Organization)] NBR [Norma Brasileira Regulamentadora (Brazilian Regulatory Standard)] 13875:2014 (ABNT, 2014), called "Agrochemicals and the

Table 1 - Trade name, formulation and doses of registered agrochemicals used for rice and/or paddy field rice culture and in tank mix for evaluation of physicochemical compatibility. Capão do Leão, RS, 2016

Treatments – Commercial product (c.p.)/ active ingredient (a.i.)	Formulation*	Dose of c.p. ha^{-1} (L or kg of c.p.)
Mixture of herbicide		
Clincher® + Ricer®(1)/cyhalofop butyl + penoxsulam	EC + CS	1.75 + 0.25
Clincher® + Kifix®(2)/cyhalofop butyl + (imazapyr + imazapique)	EC + WG	1.75 + 0.14
Clincher® + Imazethapyr Plus Nortox®(1)/cyhalofop butyl + imazethapyr	EC + SL	1.75 + 1.00
Clincher® + Ricer® + Kifix®(2)/cyhalofop butyl + penoxsulam + (imazapyr + imazapique)	EC + CS + WG	1.75 + 0.25 + 0.14
Clincher® + Ricer® + Sirius® 250 CS(1)/cyhalofop butyl + penoxsulam + pyrazosulfuron-ethyl	EC + CS + CS	1.75 + 0.25 + 0.08
Imazethapyr Plus Nortox® + Basagran® 600(2)/imazethapyr + bentazone	SL + SL	1.00 + 1.60
Mixture of herbicide and insecticide		
Clincher® + Ricer® + Arrivo® 200 EC(1)/cyhalofop butyl + penoxsulam + cypermethrin	EC + CS + EC	1.75 + 0.25 + 0.75
Clincher® + Kifix® + Arrivo® 200 EC(2)/cyhalofop butyl + (imazapyr + imazapique) + cypermethrin	EC + WG + EC	1.75 + 0.14 + 0.75
Clincher® + Imazethapyr Plus Nortox® + Arrivo® 200 EC(1)/cyhalofop butyl + imazethapyr + cypermethrin	EC + SL + EC	1.75 + 1.00 + 0.75
Clincher® + Ricer® + Kifix® + Arrivo® 200 EC(2)/cyhalofop butyl + penoxsulam + (imazapyr + imazapique) + cypermethrin	EC + CS + WG + EC	1.75 + 0.25 + 0.14 + 0.75
Clincher® + Ricer® + Sirius® 250 CS + Arrivo® 200 EC(1)/cyhalofop butyl + penoxsulam + pyrazosulfuron-ethyl + cypermethrin	EC + CS + CS + EC	1.75 + 0.25 + 0.08 + 0.75
Imazethapyr Plus Nortox® + Basagran® 600 + Arrivo® 200 EC(2)/imazethapyr + bentazone + cypermethrin	SL + SL + EC	1.00 + 1.60 + 0.75
Mixture of fungicide		
Alterne® + Bim® 750 BR + Priori® 250 CS/tebuconazole + tricyclazole + azoxystrobin	EC + WP + CS	0.75 + 0.30 + 0.40
Mixture of fungicide and insecticide		
Bim® 750 BR + Actara® 250 WG/tricyclazole + thiamethoxam	WP + WG	0.30 + 0.15
Alterne® + Bim® 750 BR + Priori® 250 CS + Actara® 250 WG/tebuconazole + azoxystrobin + thiamethoxam	EC + WP + CS + WG	0.75 + 0.30 + 0.40 + 0.15
Alterne® + Bim® 750 BR + Priori® 250 CS + Talisman®/tebuconazole + tricyclazole + azoxystrobin + (bifenthrin + carbosulfan)	EC + WP + CS + EC	0.75 + 0.30 + 0.40 + 0.30

* EC: emulsifiable concentrate; WP: wettable powder; CS: concentrated suspension; SL: soluble (liquid) concentrate; WG: water dispersible granules. (1) Addition of vegetable oil to the spray mix (0.5% v/v); (2) Addition of mineral oil to the spray mix (0.5% v/v).

like –physicochemical compatibility assessment," following a dynamic technique, that is, by stirring.

Prior to mixing, agrochemicals were individually tested for their dispersion characteristics (homogenization) in the spray mix. Spray mixes were prepared using standard water with a total hardness of 20 mg kg^{-1} in $CaCO_3$ as a vehicle, according to NBR 13074:2004 (ABNT, 2004), and the maximum doses recorded for each agrochemical (AGROFIT, 2016) in a volume of application of 200 L ha^{-1}.

Agrochemicals in liquid and solid formulations were dosed by means of an automatic pipette and an analytical balance, respectively. First, 150 mL of standard water were added to a graduated cylinder with lid and after the addition of each agrochemical the lidded graduated cylinder was inverted, rotated at 180 °, and the movement were repeated ten times (once every two seconds) in order to obtain good homogenization. Addition of the products occurred in the following sequence: first the solid formulations were added, in the sequence WG (water dispersible granules) and WP (wettable powder). Subsequently, the liquid formulations were added, in the sequence CS (concentrated suspension), SL (soluble concentrate), and EC (emulsifiable concentrate). After the last addition, the graduated cylinder was filled with standard water to the 250 mL mark, repeating the procedure of inverting the graduated cylinder ten times. Finally, the spray mix was transferred to a 500 mL beaker and kept under constant stirring for two hours by means of a mechanical magnetic stirrer.

The appearance of the spray mix was visually evaluated, according to the presence (P) or absence (A) of the following parameters: homogeneity, flocculation, sedimentation, phase separation, lump formation by sieve with nominal opening 149 µm, according to NBR NM ISO 3310-1:2010 (ABNT, 2010), separation of oil, formation of crystals, cream and foam. The pH of the spray mix was measured using a (pocket type) portable pH meter of known accuracy of ± 0.1 pH.

It was possible to observe physical compatibility in the mixtures among herbicides, between herbicide and insecticide, among fungicides, and between fungicide and insecticide, with absence of flocculation, sedimentation, phase separation, lumps, oil separation, formation of crystals, cream and foam (Table 2). These physical characteristics indicate that the influence on spray formation mechanisms, such as flow, formation, drop distribution, and volumetric distribution pattern, shall be minimal, resulting in treatments with higher efficiency and lower drift and environmental contamination risks (Miller and Butler Ellis, 2000; Petter et al., 2013).

Regarding the pH verified after stirring the spray mixes, the values ranged between 2.19 and 6.89. In general, mixing different products to the spray mix did not exert too much influence on the pH values considered ideal for each agrochemical. Herbicides efficiency can be increased when the pH is in values close to 5.0 (Stock and Briggs, 2000). However, the association of herbicide Kifix® with different agrochemicals significantly interfered in the spray mix pH, making it more acidic, with values ranging between 2.19 and 2.22. These values may influence the treatment efficiency in the biological target and interfere with the dissociation levels of the active ingredients contained in the spray mix (Kissmann, 1998). Spray mix acidification reduces the molecules dissociation. Thus, herbicides dissolved under low pH conditions shall be more easily absorbed by plants because the molecules are in a non-dissociated form (Wanamarta and Penner, 1989).

The absence of physical incompatibility among the mixtures of agrochemicals in the present study can be attributed to the correct sequence of addition of the agrochemicals and the long period of stirring the mixture, specified in NBR 13875:2014 (ABNT, 2014). However, it is known that in the field most of the time farmers do not have the knowledge regarding the correct sequence for mixing agrochemicals and neither this large amount of time for stirring the spray mix, which can result in incompatibilities. Gazziero (2015) has reported about farmers' statements on observing incompatibilities involving different classes of agrochemicals when the possibility of some reaction was not investigated or when in the absence of correct orientation. The antagonistic effect of some compounds in mixture, for example, may be a consequence of physical incompatibility caused by mixing formulations WG com CS (Thiesen and Ruedel, 2004).

In this sense, prior to application it is important to observe the limitations of use contained in package inserts regarding the use of agrochemicals in the mixture in order to avoid

Table 2 - Physicochemical compatibility of mixtures of different agrochemicals registered for rice and/or paddy field rice cultivation and tested according to criteria by ABNT [Associação Brasileira de Normas Técnicas (Brazilian National Standards Organization)] NBR [Norma Brasileira Regulamentadora (Brazilian Regulatory Standard)] (ABNT NBR 13875:2014). Temperature: 25 ± 1 ºC; RH: 55 ± 10%. Capão do Leão, RS, 2016

Commercial product	pH	ho	fl	sd	ps	lu	ol	ct	cm	fo	lu*
Mixture of herbicide											
Clincher® + Ricer®(1)	5.12	P	A	A	A	A	A	A	A	A	A
Clincher® + Kifix®(2)	2.20	P	A	A	A	A	A	A	A	A	A
Clincher® + Imazetapir Plus Nortox®(1)	6.27	P	A	A	A	A	A	A	A	A	A
Clincher® + Ricer® + Kifix®(2)	2.19	P	A	A	A	A	A	A	A	A	A
Clincher® + Ricer® + Sirius® 250 CS(1)	5.25	P	A	A	A	A	A	A	A	A	A
Imazetapir Plus Nortox® + Basagran® 600(2)	6.36	P	A	A	A	A	A	A	A	A	A
Mixture of herbicide and insecticide											
Clincher® + Ricer® + Arrivo® 200 EC(1)	5.02	P	A	A	A	A	A	A	A	A	A
Clincher® + Kifix® + Arrivo® 200 EC(2)	2.22	P	A	A	A	A	A	A	A	A	A
Clincher® + Imazetapir Plus Nortox® + Arrivo® 200 EC(1)	5.96	P	A	A	A	A	A	A	A	A	A
Clincher® + Ricer® + Kifix® + Arrivo® 200 EC(2)	2.19	P	A	A	A	A	A	A	A	A	A
Clincher® + Ricer® + Sirius® 250 CS + Arrivo® 200 EC(1)	5.17	P	A	A	A	A	A	A	A	A	A
Imazetapir Plus Nortox® + Basagran® 600 + Arrivo® 200 EC(2)	3.25	P	A	A	A	A	A	A	A	A	A
Mixture of fungicide											
Alterne® + Bim® 750 BR + Priori® 250 CS	3.74	P	A	A	A	A	A	A	A	A	A
Mixture of fungicide and insecticide											
Bim® 750 BR + Actara® 250 WG	6.89	P	A	A	A	A	A	A	A	A	A
Alterne® + Bim® 750 BR + Priori® 250 CS + Actara® 250 WG	3.83	P	A	A	A	A	A	A	A	A	A
Alterne® + Bim® 750 BR + Priori® 250 CS + Talisman®	6.55	P	A	A	A	A	A	A	A	A	A

P: presence or A: absence (of the evaluation criteria). pH after stirring; ho: homogeneity; fl: flocculation; sd: sedimentation; ps: phase separation; lu: presence of lumps; ol: oil separation; ct: crystal formation; cm: cream; fo: foam; lu*: presence of lumps (sieve of 149 µm). (1) Addition of vegetable oil to spray mix (0.5% v/v); (2) Addition of mineral oil to the spray mix (0.5% v/v).

incompatibilities. In addition, a premix is recommended to check for undesired reactions (Gazziero, 2015).

Therefore, mixtures of agrochemicals under stirring among herbicides (Clincher® + Ricer®, Clincher® + Kifix®, Clincher® + Imazethapyr Plus Nortox®, Clincher® + Ricer® + Kifix®, Clincher® + Ricer® + Sirius® 250 CS, and Imazethapyr Plus Nortox® + Basagran® 600), between herbicide and insecticide (Clincher® + Ricer® + Arrivo® 200 EC, Clincher® + Kifix® + Arrivo® 200 EC, Clincher® + Imazethapyr Plus Nortox® + Arrivo® 200 EC, Clincher® + Ricer® + Kifix® + Arrivo® 200 EC, Clincher® + Ricer® + Sirius® 250 CS + Arrivo® 200 EC and Imazethapyr Plus Nortox® + Basagran® 600 + Arrivo® 200 EC), among fungicides (Alterne® + Bim® 750 BR + Priori® 250 CS) and among fungicides and insecticides (Bim® 750 BR + Actara® 250 WG, Alterne® + Bim® 750 BR + Priori® 250 CS + Actara® 250 WG and Alterne® + Bim® 750 BR + Priori® 250 CS + Talisman®) do not alter the physical characteristics of the spray mix, being compatible for use in mixing in the spray tank in plant treatments in paddy field rice crops.

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