

CONTROLLED RELEASE FORMULATIONS OF ATRAZINE PRODUCED BY TUMBLING AGGLOMERATION

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Abstract: The herbicide atrazine was incorporated in the granules manufactured in the process of tumbling agglomeration to obtain controlled release (CR) formulations. The formulations contained bentonite as a CR matrix forming agent (960–974 g/kg of dry granules), atrazine (10 g/kg), citric acid (3.2 g/kg), and sodium alginate as a matrix binder and a release modifier (12.8–26.8 g/kg). The release characteristics of atrazine were studied by immersion of the granules in static water. The effects of formulations on atrazine transport through soil were studied using model soil columns irrigated with water. The release of atrazine from CR granules into water was affected by increasing the alginate concentration in a particular formulation because the time necessary for the release of 50% of the active ingredient was longer for the granules containing a higher amount of alginate. The CR formulations significantly reduced the amount of atrazine leached to the soil surface horizon in comparison with the commercial water suspension of the herbicide.

Key words: controlled release, herbicide, atrazine, alginate, tumbling agglomeration

INTRODUCTION

Among all pesticides herbicides such as atrazine are applied in relatively large amounts. Herbicide formulations that are currently used are principally solutions, liquid suspensions, emulsifiable concentrates, and wettable or flowable powders. The active ingredient in these formulations is readily available for leaching, runoff or volatilization losses on application. Losses of an active ingredient of 9 to 12% from dispersible granules and wettable powder formulations, and 4 to 8% from emulsion and dispersible liquid formulations have been reported (Wauchope et al. 1990). For this reasons, to ensure adequate pest control for a sufficient period of time, herbicides are used in amounts greatly exceeding those required for the target organism control. These excessive quantities of herbicides used in certain sectors of agriculture increase the possibility of runoff or leaching and thus increase contamination of sur-

face and ground water (Holden et al. 1992), which in turn leads to the potential development of certain types of tumors in humans (Van Leeuwen et al. 1999).

Controlled release (CR) formulations may provide a new and environment friendly alternative to the conventional herbicide formulations. CR formulations differ from the conventional formulations in that only a part of the active ingredient is available in the soil at any given time. The bulk of the herbicide is trapped in an inert formulation matrix at or near soil surface and is less subjected to leaching to the water sources (e.g. atrazine), degradation (e.g. alachlor), or runoff and volatilization losses (Vasilakoglu et al. 1997).

The advantages of CR formulations are: fewer chemicals required for weed control, a less risk of environmental pollution, savings in the manpower and energy due to fewer applications required in comparison to the conventional formulations, increased safety for the farmer or herbicide user, and fewer non-target effects (Małyszka and Jankowski 2000).

Much of the work carried out to date on CR systems has focused on formulations using matrix forming substrates such as starch (Carr et al. 1991, Chen and Jane, 1995), kraft lignin (Cotterill and Wilkins 1996), and clay minerals (Fernandez-Perez et al. 2000; Gerstl et al. 1998). Methods based on thermo-mechanical (Schreiber et al. 1993) and thermo-chemical (Ferraz et al. 1997) treatments of matrix components were used for the production of CR formulations. CR matrices were also manufactured by the encapsulation method utilizing sodium alginate as a gelling agent (Johnson and Pepperman 1996, Pepperman and Kuan 1993). The use of thermal methods is not acceptable for temperature sensitive active ingredients (e.g. alachlor), while the encapsulation method is difficult and expensive when scaling up of the process is considered.

The tumbling agglomeration can be an alternative method avoiding the disadvantages of the current procedures of CR formulation manufacture. In this method an inclined pan of a diameter ranging from 0.4 to 7.5 m rotates a powder to be granulated (Snow et al. 1973). The particles of feed material are moistened by spraying a liquid onto the tumbling load. In this process aggregates are formed by wet particles sticking together. Physical properties of granules formed by the tumbling granulation, such as granule size and shape, porosity, mechanical strength, and solubility depend on several operating variables including liquid content and residence time, as well as the addition of binders to the moisturizing liquid (Klassien and Griszajew 1989).

In the present work, tumbling agglomeration is proposed as a new method for the manufacturing of controlled release formulations of the common herbicide atrazine. Some physical properties of the obtained CR formulations were characterized and the release rate of herbicide from CR granules was studied using a static immersion test and soil column tests, and compared to that of commercial formulation.

MATERIALS AND METHODS

Chemicals: Bentonite (Bentonit Special, Zębic S.A, Poland) of 12% moisture content was used as the CR matrix forming material. This is a clay mineral with a chemical formula $(Al,Mg)_2[Si_4O_{10}(OH)_2] \cdot Ca_{0.2} \times (H_2O)_x$, which contains about

70% of montmorillonite. Sodium alginate (Manucol DM, Kelco, San Diego, U.S.A.) was used as a CR matrix binding agent. Powdered atrazine [2-chloro-4-ethylamino-6-isopropylamino-s-atrazine], technical grade (90% active agent) and granular formulation of the atrazine (Gesaprim 90WG) were supplied by Novartis Crop Protection AG (Basel, Switzerland). Solvents used in the mobile phase for HPLC determinations were: HPLC grade acetonitrile (Merck, Darmstadt, Germany) and milliQ quality water (Millipore). Other materials used were analytical grade atrazine (Fluka) used as an external standard, citric acid, methanol, and house-supplied distilled water.

Soil: The soil used for column studies was the surface horizon (0–22.5 cm) pseudopodzol soil from the Agricultural University Experimental Station at Złotniki. The soil is classified as loamy sand, and contains 1.1% of organic matter. The soil was air-dried and checked for residues of atrazine by extraction with methanol and HPLC analysis. No residues were found.

Preparation of controlled release formulations: The CR formulations of atrazine were obtained by the tumbling agglomeration of the bentonite powder, taking into account the crosslinking properties of the alginate in the presence of divalent cations. A laboratory inclined-pan agglomerator (50 deg from horizontal), consisting of a rotating pan (0.4 m diameter) with a rim (0.08 m) and a vertical scraper was filled with preblended bentonite powder with atrazine and various amounts of alginate powder. Table 1 shows the amounts of components used per kilogram of dry granules. The pan was rotated at 30 rpm causing the powder to lift and cascade. The moving material was periodically sprayed with 1% (w/v) water solution of citric acid (50 ml) from the nozzle located in the upper part of the pan. After a sufficient residence time, spherical granules were removed from the pan, then air-dried (40°C) to a constant weight and analyzed for particle size.

Sieve analysis of granulates: Particle size distribution of granulates was examined using the sieve analysis. For these experiments a set of screens with a mesh size of 5, 2.5, 1.5 and 1.0 mm was used. The granules were placed on the top sieve with the largest mesh size and shaken for 5 minutes. Data from these experiments were converted to the volumetric mean size (d_v) with the following formula:

$$d_v = \sqrt[3]{\frac{\sum x_i}{\sum \frac{x_i}{d_i^3}}}$$

where: x_i – mass fraction of the sample on a particular screen; d_i – mean mesh size of two adjoining screens.

Static immersion test: An accurately weighed amount of dry CR granules (10 mg) was immersed in distilled water (100 mL, pH 5.5–6.0) at 25°C with three replicates for each formulation. Samples (1 mL) were taken for HPLC analysis at different time intervals. After each sampling time all water was replaced with fresh water to minimize saturation effects. The aliquots from the static immersion tests

Table 1. Amounts of components used in controlled release formulations (dry granules)

Formulation	Bentonite (g/kg)	Alginate (g/kg)	Atrazine (g/kg)	Citric acid (g/kg)
CRF 1	974	12.8	10	3.2
CRF 2	968	18.8	10	3.2
CRF 3	960	26.8	10	3.2

(1 mL) were diluted 1:4 with methanol and the resulting extracts were filtered through a siringe filter (0.45 μm), and stored at 4°C prior to HPLC analysis.

Soil column tests: Air-dried soil was ground to pass a 1.0 mm sieve and then packed into Plexiglass pipes (0.12 m i.d. and 0.30 cm length) to a depth of 25 cm. The bulk density of the soil was 1.3 g/cm³. Prior to the application of herbicide treatments, the columns were saturated with tap water to a field capacity and allowed to equilibrate for 24 h. A commercial, granular formulation of atrazine was dissolved in tap water according to the manufacturer's instructions to make the concentration of the active ingredient of 8.3 g/L and applied to the soil surface of a control column with a hand sprayer at a rate equivalent to 1.6 kg a.i./ha. Prewighed amounts of CR formulations were mixed uniformly with a top 1 cm of the soil in the other three columns to obtain the atrazine concentration equivalent to 1.6 kg a.i./ha. Simulated rainfall generated by a hand sprayer was applied to the soil surface in three equal doses at 10 day intervals to obtain a total of 49 mm (average for May rainfall in the region). Following water treatment, the soil surface was covered with Parafilm to minimize evaporation and herbicide volatilization. After 5 weeks, the soil was extruded from the columns and divided into 5 cm sections for extraction and analysis of atrazine. All sections were air-dried and subsamples (5g) from each section were extracted with methanol (soil to methanol ratio of 1:2). The methanol extracts from the soil column tests were filtered through Whatman #4 filter paper and then through 0.45 μm filter and stored at 4°C prior to HPLC analysis.

Chromatographic analysis of atrazine: Analysis of atrazine was carried out on the HPLC (LaChrom, Merck/Hitachi) equipped with a ODS-Hypersil column (Hewlett Packard). The HPLC operating conditions were as follows: sample volume 40 mL; flow rate 0.5 mL/min; and the mobile phase, a 35:65 water/acetonitrile mixture at 25°C. Atrazine was analyzed at 222 nm, its wavelength of maximum absorption. Three replicates were made for each extract.

RESULTS

Tumbling agglomeration of powdered components of CR atrazine formulations resulted in the formation of spherical granules with a non-uniform size distribution, where the largest mass fraction produced was that of the range of diameter 1.0–2.5 mm (Tab. 2). Increasing alginate concentration in CR formulation led to an increase of the amount of larger size granules and thus an increase of the overall mean size of agglomerates.

The release studies of atrazine into static water were limited to the fraction of 1.0–2.5 mm. As can be seen from figure 1, all CR formulations showed a decline in the release of herbicide with time, while the release rates were different and strongly depended on alginate concentration. The release data were analysed by ap-

Table 2. The influence of alginate concentration on the particle size distribution of CR formulations

Particle size range (mm)	Mass fraction (%)		
	Formulation		
	CRF 1	CRF 2	CRF 3
> 5.0	2.2	3.7	4.2
2.5–5.0	18.8	26.8	30.7
1.0–2.5	74.5	67.2	63.9
< 1.0	4.5	2.3	1.2
d_v (mm)	1.26	1.47	1.65

plying the generalized model proposed by Ritger and Peppas (1987) for the diffusion-controlled matrix systems:

$$\frac{M_t}{M_o} = k \cdot t^n$$

where M_o is the initial active-agent loading of the matrix, M_t is the quantity of the active agent released after time t , and k and n are the system parameters that depend on the nature of matrix-penetrant-active agent interaction. The data were fitted up to 70% release of the herbicide. The release profiles of atrazine from CR formula-

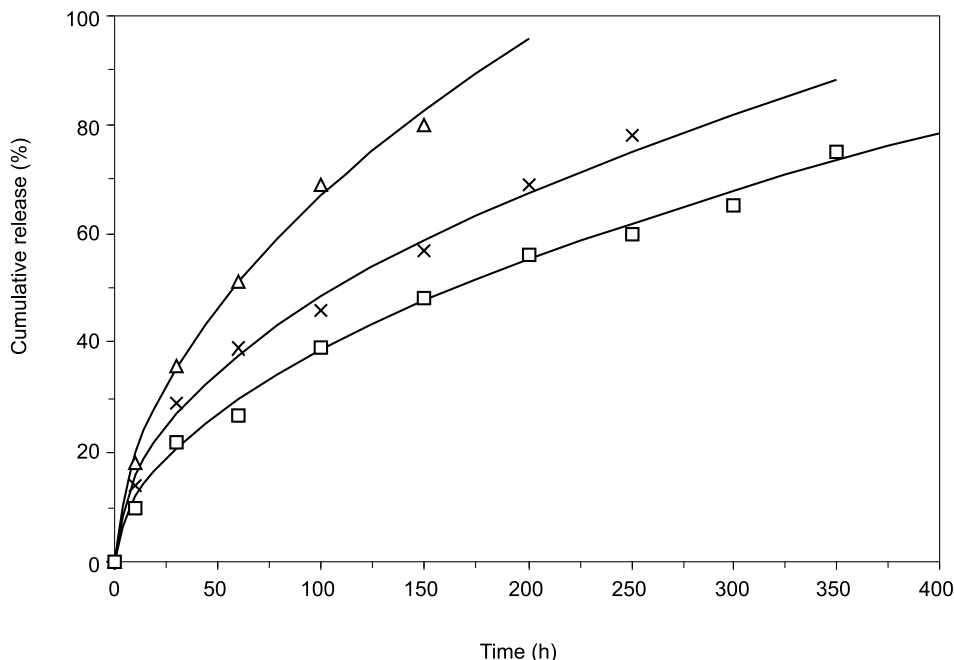


Fig. 1. Rate of release of atrazine from CR granular formulations into static water: Δ – CRF 1, \times – CRF 2, \square – CRF 3

Table 3. Constants for the release data of atrazine from CR formulations into static water

CR formulation	k^* (h^{-n})	n^*	r^2	T_{50} (h)
CRF 1	0.061 (0.006)	0.52 (0.02)	0.992	57
CRF 2	0.053 (0.008)	0.48 (0.01)	0.998	107
CRF 3	0.037 (0.004)	0.51 (0.02)	0.996	165

*standard error in parentheses; r – correlation coefficient; T_{50} – time required to release 50% of atrazine

tions fit well to the release model and the values of k and n together with the correlation coefficients are shown in table 3. The release rates of atrazine into static water were different for the investigated CR formulations and decreased with the increase of alginate concentration in the granules while the n values ranged from 0.48 to 0.52. From the obtained constants given in table 3, the T_{50} values were calculated (time taken for 50% of the atrazine to be released). T_{50} was found to increase with alginate concentration in the CR formulations, ranging from 57 to 165 hours.

Atrazine distribution in the soil columns 5 weeks after application and simulated rainfall is presented in figure 2. Atrazine applied as commercial formulation displayed considerable mobility in soil and significant amounts were observed as deep as 20 cm. On the other hand, atrazine applied as CR formulation was leached from the granules at a lower rate and was detected mostly in the surface layers of the soil columns. The relationship between the amount of atrazine leached and alginate concentration in CR formulations was similar to that observed in static immersion tests.

Depth (cm)

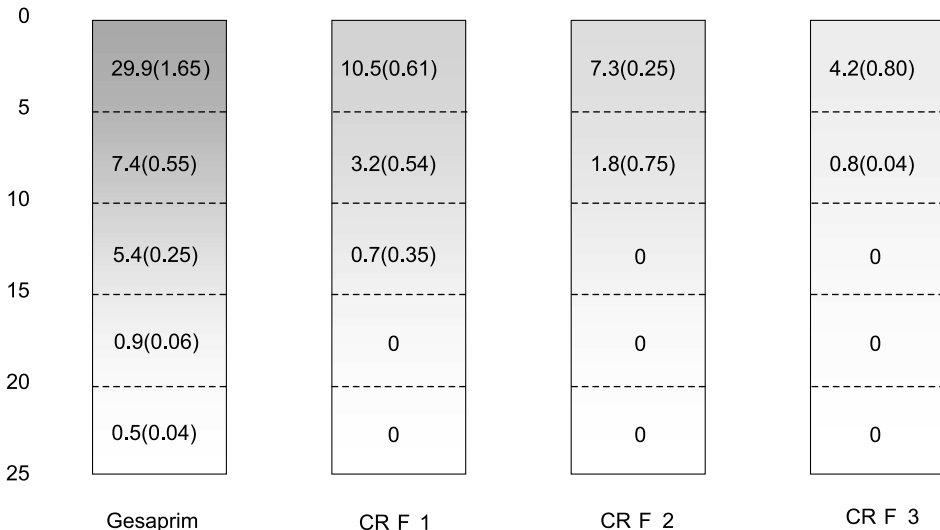


Fig. 2. Distribution of atrazine (mg/kg) in soil columns 5 weeks after application as a commercial formulation or as CR formulation (standard error in parentheses)

DISCUSSION

The process of tumbling agglomeration widely applied for granulation of fertilizers involves the formation of aggregates on the basis of Van der Waals forces, electrostatic attraction and the creation of chemical bonds (Snow et al. 1973). In this study tumbling agglomeration was used to manufacture CR formulation of atrazine incorporating a natural polymer – alginate into bentonite matrix. Bentonite, a clay mineral, has some atrazine-binding capability and contains magnesium and calcium salts (Gilchrist et al. 1993). In an acidic environment bentonite releases cations (Ca^{2+} and Mg^{2+}) which are known as promoters of crosslinking reaction between alginate molecules (Grant et al. 1973). Thus, alginate used in the granulation process served as a matrix binder that could influence integrity and solubility of the granules containing atrazine. This polymer has frequently been applied in preparations of agricultural CR formulations by encapsulation of various herbicides using the drop-forming technique (Johnson and Pepperman 1996, Gerstl et al. 1998, Fernandez-Perez et al. 2000). In this procedure the formulation water mixture containing kaolinite, bentonite or montmorillonite as matrix fillers, herbicide, and alginate is dropped into a calcium chloride solution to form spherical gelled beads which are subsequently dried. The addition of clay to the alginate gel is expected to decrease the rate of release of the trapped active compound both as a result of sorption of the compound and due to increased tortuosity within the gel.

The granules obtained in this work by the tumbling agglomeration were spherical but non-uniform in size. Since the size of CR formulation has a profound effect on the rate of release of active ingredient (Gerstl et al. 1998), this appears to be the main disadvantage in comparison with the encapsulation technique used previously for the preparation of CR herbicide granular formulations, where the granules were equal in size (Johnson and Pepperman 1996). However, other procedures evaluated for the preparation of CR formulations such as starch extrusion (Carr et al. 1991) or lignin entrapment (Cotteril and Wilkins 1996) also produced non-uniform granules. In a scaled-up version of the present method, a uniform product would be obtained using a continuously working agglomeration device with a constant feed of raw material and a constant flow rate of the moisturising solution.

The n values of the empirical model close to 0.5 indicated that the release of herbicide was controlled by a diffusion mechanism. According to Smith and Herbig (1993) in diffusion-controlled matrix systems the amount of the released ingredient is proportional to the square root of time. This results from the increasing distance through which the molecules have to diffuse as the depleted zone advances to the center of the spherical granule. A similar, diffusion-controlled mechanism of release was observed by Ferraz et al. (1997) for 2,4-D herbicide from six lignin based granular formulations, and by Gerstl et al. (1998) for alachlor and atrazine from clay-alginate-pectin formulations in water immersion experiments. On the other hand, Cottrill and Wilkins (1996) evaluated the release of five phenylurea herbicides entrapped into a kraft lignin matrix system and reported the values of n in the range of 0.21 to 0.61. These deviations could indicate interactions of matrix components with the diffusion agent depending on physicochemical properties of a particular herbicide.

From the data presented in table 2 it is evident that the release rate of atrazine was influenced by alginate concentration in a particular formulation. The T_{50} , a parameter frequently used by various authors to compare the CR formulations, was in the present work longer for the granules containing greater alginate concentration. Similar effect caused by increasing the polymer concentration in the alginate microparticles was observed by Kim and Lee (1992) for the release of blue dextran. It is assumed that diffusivity decrease in the matrices containing polymer is proportional to its crosslinking density since the polymer network creates obstacles for a diffusing molecule, giving a tortuous increased diffusional path (Smith and Herbig 1992).

T_{50} values reported in the literature for several slow-release formulations of herbicides significantly varied depending on the type of formulation and the experimental conditions used. Gerstl et al. (1998) compared the release characteristics of atrazine and alachlor from alginate and pectin encapsulated formulations containing kaolinite and montmorillonite into stirred water. They reported T_{50} varying from 7 h for alachlor-pectin formulation to 12 days for atrazine-alginate-montmorillonite capsules. Fernandez-Perez et al. (2000) studied the release of diuron and atrazine into still water from pure alginate and alginate-bentonite-humic acid CR formulations reporting T_{50} values ranging from 10 to 50 days. A lignin matrix herbicide formulations were also reported to exhibit excellent release control over a wide range of T_{50} values varying from several days to 15 weeks (Cotteril and Wilkins 1996; Ferraz et al. 1997).

The mobility of atrazine from CR formulations obtained by the tumbling agglomeration was compared to that of a commercial liquid formulation in soil columns experiments. It was shown that CR formulations retained a greater amount of the applied atrazine in the soil surface, while the application of liquid suspension of atrazine resulted in a high concentration of herbicide in the application zone but also quite significant leaching to depths of over 20 cm. The decreased leaching potential observed for CR formulations and retention of atrazine in the surface horizon is desirable for a number of reasons. First, it improves the efficacy of weed control in this zone. Second, the herbicide is more likely to be degraded in this biologically active zone than in subsoils. Third, the use of CR formulations can also reduce the downward transport of herbicide to shallow ground water thus decreasing the risk of the environmental pollution.

Several researchers have demonstrated increased surface concentration of herbicides applied as CR formulations both in a model and in field experiments. For example, Johnson and Pepperman (1995) found that alginate-kaolin-oil encapsulated formulations retained from 58 to 93% of the applied atrazine in the soil surface in column experiments. Moreover, Mervosh et al. (1995) studied clomazone and atrazine transport through soils applying starch-encapsulated and technical-grade herbicides. They found that CR formulations would retain 41% of clomazone and 85% of atrazine in the top 7 cm of soil compared to 34% and 21% when applied as technical material.

CR granular formulations obtained by the tumbling agglomeration studied in this work appear to hold promise as a herbicide management system. Our results suggest that the use of bentonite as a matrix building agent and alginate as a release

rate controlling agent could reduce leaching of atrazine in comparison with a technical-grade herbicide. Considering simplicity of the technique used to produce CR formulations and the easiness of the scaling-up procedure, formulations with the desired release profile can readily be made by varying in the granule size, the percentage of herbicide and the inclusion of additives.

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POLISH SUMMARY

PREPARATY ATRAZYNY O KONTROLOWANYM UWALNIANIU WYTWARZANE ZA POMOCĄ GRANULACJI TALERZOWEJ

Atrazynę unieruchomiono w granulacie wytworzonym za pomocą granulacji talerzowej w celu otrzymania postaci herbicydu o kontrolowanej szybkości uwalniania. Granulat zawierał bentonit (960–974 g/kg suchego preparatu), atrazynę (10 g/kg), kwas cytrynowy (3,2 g/kg) oraz alginian sodu (12,8–26,8 g/kg) spełniający rolę substancji wiążącej składniki matrycy i modyfikującej szybkość uwalniania aktywnego składnika. Charakterystykę uwalniania atrazyny zbadano metodą immersyjną w wodzie, w warunkach statycznych. Zbadano także przenikanie atrazyny do gleby w modelowych kolumnach glebowych zraszanych wodą w celu symulacji opadu atmosferycznego. Szybkość wypływu atrazyny z granulatu zmniejszała się wraz ze wzrostem zawartości alginianu w matrycy, a czas połówkowego wypływu atrazyny odpowiednio się wydłużał. Unieruchomienie atrazyny w granulacie o kontrolowanym uwalnianiu spowodowało zmniejszony wpływ herbicydu do górnej warstwy gleby, w porównaniu z handlowym preparatem zastosowanym w postaci wodnej zawiesiny.