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Glycerine associated molecules with herbicide for controlling *Adenocalymma peregrinum* in cultivated pastures

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The weed *Adenocalymma peregrinum* that is popularly known in Brazil as “ciganinha”, belongs to the family Bignoniaceae. The only way to control this plant species in crop fields is by the application of herbicides on the stump or directly on the stem. The present study aimed to analyze the effect of glycerine in controlling *A. peregrinum* (MIERS) L.G.Lohmann when applied on the stem. The glycerin used as a spray application of herbicide, underwent pre-purification processes with different concentrations of phosphoric acid (85%) and was characterized for water content, sulphated ash, total glycerol, matter organic non-glycerol (MONG), methanol and pH. For the analysis of the chemical composition of the stem lignin, holocellulose, extractives, calorific value and elementary quantitative determination of C, N, H, S were determined, as well as the total content of oxygen from the stem. The field work was installed at the town of Alvorada-TO, following the randomized complete block design with six treatments and four replications. Results show that the stem of *A. peregrinum* contains a significant amount of nitrogen, compared to other species, and high lignin content which makes it the most resistant species. The use of glycerin combined to the herbicide (picloram and triclopyr), was not efficient when compared to diesel oil. It was observed that the glycerin has potential as a vehicle for applying herbicides, leaving much to the development of new studies to make changes in its physical-chemical characteristics.

Key words: Weeds, pastures, management, *Adenocalymma peregrinum*.

INTRODUCTION

Livestock is one of the most important economic activities in Brazil, accounting for 8.9% of national GDP. According to Harris (2010), degradation of pastures is a major problem in Brazilian cattle. About 70 million hectares of pastures in the Central West and Northern Brazil have been degraded or are undergoing degradation. This

occurs due to low soil fertility, inadequate forage, high grazing pressure, and mainly due to high infestation weeds (Feldman and Refi, 2006). A plant species is considered a weed if it is growing in places where it is detrimental to human activities such as interfering with the development of commercial crops or poisoning

livestock (Christoffoleti et al., 2004). Among the weeds that are more difficult to control are shrubs, in which the most important are those with vegetative reproductive structures. In areas of savanna *Adenocalymma peregrinum*, popularly known in Brazil as “Ciganinha” stands out. It is in the family Bignoniaceae, is native to the Cerrado of Brazil, tropical savanna ecoregion, and has become invasive in cultivated pastures (Christoffoleti et al., 2004), and is very difficult to control (Grassi et al., 2005). Among the molecules used in controlling shrubby and semi-shrubby weeds are the acid 3,5,6-trichloro-2-pyridyloxyacetic acid (triclopyr) and 4-amino-2,3,5,6-tricloropiridina-carboxylic acid (picloram), which are the basics active major herbicides registered for pasture since these molecules and selectively systemic, belonging to the class of compounds that has a pyrimidine ring as the base structure. Together, these molecules form the herbicide (picloram and triclopyr), which is recommended for the control of annual and perennial broadleaf pasture weeds. Application of herbicide is commonly made with diesel oil, which is a very costly product and presents a high toxicity to the environment. Therefore, it is necessary to replace this with a safer and cheaper product.

One possibility to improve the efficiency of chemical control of weeds is the association of herbicides with adjuvants (Banks et al., 2014). This procedure may improve the efficiency of the herbicide by acting on the molecular structure of the solution. As a result, it breaks the surface tension, adds energy to break molecular bonds by acting directly in the plant tissue, which facilitates and assists the cuticular penetration (Banks et al., 2014). A promising product to be used as adjuvant is the glycerin that is obtained from biodiesel transesterification reaction. It has been produced on a large scale, low cost and has a low potential for evaporation. The hypothesis of this study is that glycerin can become a solvent for application of herbicide molecules. Therefore, it is necessary to know the physicochemical properties of glycerine and the chemical composition of the weed stem *A. peregrinum*.

This study aimed to evaluate the effect of glycerin in replacement of diesel in controlling *A. peregrinum* in cultivated pastures.

MATERIALS AND METHODS

The methodologies used in the tests are grouped into three stages: Chemical analysis of the stem of *A. peregrinum*; evaluation of physical-chemical parameters of glycerin samples and evaluation of herbicide efficiency in controlling *A. peregrinum* in the field using glycerin as pre-purified spray application.

Chemical analysis of the stem of *A. peregrinum*

Stems from the weed species *A. peregrinum* were collected in July 2011 in an area belonging to the municipality of Alvorada, Tocantins, located in the geographical coordinates 12° 28' 48" south latitude and 49° 07' 29" longitude West Greenwich, the 280 m (two hundred and eighty meters of altitude above mean sea level). The collected plants contained stems with 3 cm in diameter, which is the recommended size for herbicide control. Samples of 3 cm in diameter and 20 cm in length were removed and oven dried in forced air circulation at a temperature of 70°C until constant weight. Later, the samples were ground in a hammer mill and passed through sieves of 40 and 60 Mesh.

Determination of lignin, holocellulose and extractives

We used a standardized methodology for ABCP (Brazilian Technical Association of Pulp and Paper; No. 1974, M70/71 M3/69 ABTCP, ABTCP, 1974). Analyses were performed in triplicate.

Determination of gross calorific value

This procedure was based on the ABNT NBR 8633/84 (ABNT, 1984), where a pump calorimeter was used (Ikar, C 200).

Elemental quantitative determination of C, N, H, S and O

Samples of stem of *A. peregrinum* were dried in air forced circulation stove at 60°C until constant weight and ground in a Wiley mill. They underwent screening in 200 mesh mash. These in turn were subjected to further dewatering in circulation oven at a temperature of 103 ± 3°C for 4 h. Finally, the samples were fractionated in accurately balance of aid, and concocted up in small envelopes to be inserted into the analyzer: Elementar® model Vario micro cube (carbon, hydrogen and nitrogen); through which it was possible for the determination of carbon, hydrogen, nitrogen, sulfur and oxygen difference of the material analyzed.

Methodology for evaluation of the physical-chemical parameters of glycerin samples

The crude glycerin was obtained from the Biotins Company, located at the municipality of Paraíso, Tocantins. The glycerin was obtained from soybean oil, which was transesterified using the catalyst NaOH (sodium hydroxide). Due to the presence of several contaminants, glycerin was previously purified with different concentrations of phosphoric acid (85%). After the addition of phosphoric acid, glycerin was placed in a separating funnel, where it remained for a rest period of 24 h for further phase separation. After the pre-purification procedure, there was the formation of three phases: the top (dark phase) presence of free fatty acids in liquid state; the intermediate stage (amber) is glycerin, and the bottom (light color) salts formed from the mixture of catalyst and phosphoric acid. Initially, an experiment was installed with eleven treatments and a control reference (distilled water) to evaluate the physical and chemical characteristics of the components of the spray solution to be used in the second part of the experiment,

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Table 1. Physico-chemical characteristics of crude glycerin.

Characteristics	Method analysis	Total
Water content (%)	ASTM E 203	2.83
Sulphated ash (%)	ASTM E D 874	9.6
Total glycerol (%)	AOCS Ea 6-94/Mod	62.14
MONG ¹ (%)	ISO 2464	25.43
Methanol	EN 14110	7.09
pH	NBR 10891	12.60

*¹Organic matter glicérica not.

performed in triplicate. The treatments consisted of: glycerin pre-purified with 5% to H₃PO₄ 85% concentration; glycerin pre-purified with 6% to H₃PO₄ 85% concentration; glycerin pre-purified with 8% to H₃PO₄ 85% concentration; glycerin pre-purified with 9% to H₃PO₄ 85% concentration; glycerin pre-purified with 5% to H₃PO₄ 85% concentration plus herbicide; glycerin pre-purified with 6% to H₃PO₄ 85% concentration plus herbicide; glycerin pre-purified with 8% to H₃PO₄ 85% concentration plus herbicide; glycerin pre-purified with 9% to H₃PO₄ 85% concentration plus herbicide; herbicide; diesel; herbicide + diesel.

The crude glycerin was characterized as the water contained, sulphated ash, glycerol, MONG, methanol and pH. These analyzes were performed by the certification possessed from laboratory, good laboratory practice by the Brazilian Institute of Metrology and Quality Technology - INMETRO. Recognition: CRL 0295 (Saybolt Concremat Technical Inspections Ltd, 2011) following rules described in Table 1. The following physical and chemical parameters of pre-purified glycerin were evaluated: viscosity, surface tension, pH, inorganic (%), acid value (mg L⁻¹), chloride content (mg L⁻¹), concentration of volatile, ash concentration (%) and impurity concentration (%). The chemical analyzes were performed according to the methodology described by the Instituto Adolfo Lutz (1985). The density is determined using the methodology described by Brazil (2010). To determine the viscosity we used a rotating viscometer analog model with EEQ-9031 Edutec range 1-20000 cP. To determine the viscosity we used a rotating viscometer analog model with EEQ-9031 Edutec range 1-20000 cP.

The surface tension was determined by the method described by Gunde et al. (2004) where we used a graduated burette and by means of the drop weight and diameter of the burette, together with the correction factor table described by Harkins and Brown (1919), it was possible to obtain the values of surface tension of the samples.

Evaluation of herbicide efficiency in controlling *A. peregrinum* in the field using pre-purified glycerin in replacement of diesel oil

The field work was installed at a production area of cattle so extensively in the city of Alvorada, Tocantins located at a latitude 12° 48 '05" and longitude 49° 12' 54". The experiment followed a randomized block design with six treatments and four replications. The experimental unit consisted of six plants of *A. peregrinum* per treatment. These plants were selected to maintaining a standard of similar age and stem diameter (3 cm). Treatments for field evaluation were: glycerin pre-purified with 5% to H₃PO₄ 85% concentration plus herbicide; glycerin pre-purified with 6% to H₃PO₄ 85% concentration plus herbicide; glycerin pre-purified with 8% to H₃PO₄ 85% concentration plus herbicide; glycerin pre-purified with 9% to H₃PO₄ 85% concentration plus herbicide; herbicide + diesel; standard commercial; control: no application. The

applications (200 mL plant⁻¹) were made at the lower third of the plant as a directed basal application around the girth until runoff, using a knapsack sprayer with a nozzle cone and without the inner core (full cone spray). Plants were evaluated for control at 30 and 60 days after application (DAA) by the percentage scale visual score, ranging from 0 to 100%, where 0 (zero) corresponds to no injury at the plant and 100 (one hundred) to the death of the plants, for considering the level of acceptance efficiency of at least 80%. At 270 DAA the number of shoots per treatment was evaluated. This method follows the rules proposed by Frans et al. (1986) and the Brazilian Society of Weed Science (1995). Data were subjected to analysis of variance by the "F" Test and the averages were compared by Tukey test at 5%. SISVAR program was used to perform the statistical analyzes.

RESULTS AND DISCUSSION

Chemical analysis of *A. peregrinum* (MIERS) L.G. Lohmann stem

The stem of *A. peregrinum* had the following chemical composition: Holocellulose (62.12%); extractives (9.32%); lignin (28.55%); carbon (46.6%); hydrogen (6.12%); carbon/hydrogen (7.61%); oxygen (45.4%); nitrogen (1.8%); carbon/nitrogen (25.8%); sulfur (0.04%); calorific value of stem of *A. peregrinum* was 4686.21 Kcal g⁻¹. Among the elements that were found, nitrogen (1.8%) was significant for *A. peregrinum*, thus taking a primary role in photosynthesis to constitute the chlorophyll molecule. The high content of ureides combined with the behavior of *A. peregrinum* as weed suggests some relation between adaptive strategies and competition with other species (Grassi et al., 2005). Among the leaves, stems and roots, the stem tends to be more lignified, which hinders the absorption of the herbicide. This was confirmed by Lacerda et al. (2006), who studied lignin concentration in shoots of *Avena byzantina* L. with analytical methods and concluded that the lignin content in the fraction was higher than in the leaf stem. According to the obtained results, it is concluded that the total extractive content corresponded to 9.32%, which may explain the adaptation of this species to limited environments. *A. peregrinum* has considerable percentage of extractives, following a standard extractive rich in other species, for example, *Quercus* spp. (10.47%), *Sassafras sassafras* L. (11.57%), *Prunus serrulata* (17.91%) *Vatairea*

Table 2. Chemical analysis of the pre-purified glycerin with 85% H₃PO₄.

Parameter	H ₃ PO ₄ (%) concentration			
	5	6	8	9
Water (%)	4.03	4.46	4.93	4.96
Acid value (mg L ⁻¹)	2.10	15.9	15.4	22.7
pH	6.20	2.32	2.28	2.01
Chloride (mg L ⁻¹)	514.46	2.033.49	2.176.80	2.621.04
Volatile at 180 °C	Absent	Absent	Absent	Absent
Inorganic (%)	18.76	13.46	24.80	37.90
Impurity (%)	0.2	0.2	0.2	0.1
Total glycerol (%)	71.09	71.52	74.13	75.97
CV %		1.50	0.82	

heteroptera Ducke (8, 88%) *Copaifera officinalis* 10.55%), *Hymenaea courbaril L.* (13.32%) *Aspidosperma tomentosum Mart.* (10.39%) *Bowdichia nitida* (8.24%) *Parapiptadenia rigida* (15.31%), and *Cariniana estrellensis*. (9.46%) (Mori et al., 2003). The calorific value is the amount of energy in the form of heat released by the combustion of a unit mass of plant which can be expressed in calories/gram or kilocalories/kilogram. According to Erol et al. (2010) combustibility and calorific value are highly influenced by the content of lignin and extractives by the presence of flammable materials (oils, resins, waxes, etc.).

Physico-chemical parameters of glycerin samples

Analyzing the physico-chemical properties of crude glycerin used in this study (Table 1), there was a glycerol content of 12.6% and a pH 62.14, which indicates the presence of a basic catalysis. The sulfated ash and material organic non-glyceric (MONG) gave values of 9.6 and 25.43%, respectively, while the residual methanol was 7.09. As expected, the increase of H₃PO₄ (85%) concentration of crude glycerine increased the acid number and expressed a concomitant reduction in pH. This had already been observed by Quispe et al. (2013), who found that with the addition of phosphoric acid at 85% concentration in the crude glycerin increasing doses actually decreased its pH and separation of the phases, for example, fatty acid salts and glycerol. The chloride and ash values (except 6%) expressed increased with the increase of phosphoric acid concentration. The increase of the doses of phosphoric acid promoted an increase in chloride content present in the glycerin. This probably is due to chemical reactions that occur in this process, since the chloride appears as a contaminant in the transesterification process for obtaining biodiesel. The concentration of glycerol increased with the addition of acid, showing the elevation of the purity of the glycerin

samples. Considering that the aim of this study was to evaluate the use of glycerin as a vehicle for spraying herbicides, it was necessary to treat it with different concentrations of phosphoric acid in order to obtain different pH levels and waste (Table 2). The following table shows the viscosity values at 40°C and surface tension corresponding to the treatments. The viscosity values of the treatments ranged from 19 to 22 cP, and treatments with pre-purified glycerin at 5 and 6% H₃PO₄ showed higher viscosity, differing significantly from the other and not between each other (Table 3). These correspond to almost five times higher than those determined in diesel and diesel mixture over herbicide. The viscosity of diesel oil checked at work is close to the technical standards described by Rossi and Ramos (2000) which is 2.05 to 4.84 cP at 40°C. Another important property of the spray for pesticide application is the surface tension resulting from the forces acting on the molecules of the liquid surface. The lower surface tension observed in treatments: diesel, herbicide (triclopyr + picloram) and herbicide (triclopyr + picloram) plus diesel ranged from 23.34 to 24.36 mNm⁻¹. Another factor to be considered in determining the viscosity of fluids is temperature. The increase of the temperature during the evaluation process decreased the viscosity, which can be explained by the molecules expansion or lower cohesion.

Butler et al. (1993) points out that, products with higher viscosity when used together with synthetic pesticides reduce evaporation and may reduce the likelihood of being washed off by rain. However, the surface tension of the droplets and their interaction with the target surface influencing not only wettability, but also the absorption process, which is critical to the effectiveness of the application (Domanska et al., 2013). One of the many functions of the surfactant consists in breaking the surface tension of the droplets surface to promote better wetting and absorption of herbicides. Analyzing the surface tension, we observed that the treatments where pre-purified glycerine was present the surface tension

Table 3. Viscosity and surface tension of the prepurified glycerin with different concentrations of phosphoric acid (85%).

Treatment	Viscosity (cP*)	Surface tension (mNm ⁻¹)
Glycerin 5%	21.95 ^a	35.43 ^b
Glycerin 6%	21.80 ^a	34.25 ^c
Glycerin 8%	20.85 ^c	35.37 ^b
Glycerin 9%	21.17 ^b	33.19 ^d
Glycerin 5% + Herbicide	20.83 ^c	30.36 ^e
Glycerin 6% + Herbicide	21.17 ^b	30.23 ^e
Glycerin 8% + Herbicide	20.43 ^d	28.45 ^f
Glycerin 9% + Herbicide	19.80 ^e	28.37 ^f
Herbicide	3,28 ^g	23.34 ^h
Diesel	4,91 ^f	23.47 ^h
Herbicide + Diesel	4.80 ^f	24.36 ^g
Distilled Water	0.99 ^h	72.40 ^a
F	4356.46**	6874.31**
CV %	1.50	0.82

*Centi-Poise. Means followed by the same letter in the column do not differ statistically from each other, according to the test Scott Knott a 5% probability; **Significant at 1%.

Table 4. Effect of herbicide in *A. peregrinum* applied to the stalk, with different enforcement vehicles.

Treatments	Percentage phytotoxicity		Shoots / treatment
	30 DAA*	60 DAA	270 DAA
Herbicide + Glycerin 5%	33.50 ^b	67.50 ^{ab}	2.75 ^c
Herbicide + Glycerin 6%	25.00 ^b	33.75 ^c	3.75 ^{bc}
Herbicide + Glycerin 8%	27.00 ^b	41.60 ^{bc}	4.50 ^b
Herbicide + Glycerin 9%	27.00 ^b	50.00 ^b	3.00 ^c
Herbicide + Diesel	84.50 ^a	88.75 ^a	1.00 ^d
Control	0.00 ^c	0.00 ^d	6.00 ^a
F	42.67**	20.85**	45.00**
DMS	19.5.	31,18	1.16
CV%	25.97	28.68	14.44

*DAA: Days after application; Means followed by the same letter in the column do not differ statistically from each other, according to the Tukey test at 5% probability; **, significant at 1%.

was greater, ranging from 28 to 36 mNm⁻¹. The treatments 5% glycerol and 8% glycerin significantly different from the others, but not between them. According to the manufacturer (Dow Agrosience, 2014) the herbicide (triclopyr + picloram) triclopyr - BUTOTÍLICO -83.53 g / l (8.353% mass / v) triclopyr acid equivalent - 60 g / l (6% mass / v), to the picloram ester, isooctyl -43.94 g / l (4.394% mass / v) equivalent acid picloram -30 g / l (3% mass / v) other ingredients -778.52 g / l (77.852% mass / v). Systemic action selective herbicide triclopyr - butotílico which belong to the chemical group piridiniloalconoico, picloram acid and the acid chemical group pyridine has surface tension of 31.58 mN m⁻¹a 25°C, higher than that found at work (23.34 mNm⁻¹)"triclopyr + picloram. This can be explained by the fact that we did not had a full temperature control

during the experiment.

Efficiency of herbicide in controlling *A. peregrinum* using glycerin in replacement of diesel oil

The effectiveness of the treatments in controlling *A. peregrinum* in the field was evaluated by scores of intoxication as well as the percentage of sprouting plants after a period of time consisting of 30, 60 DAA, DAA 270 and to the regrowth (Table 4). The treatment that simulated the standard recommendation: diesel with herbicide (tryclopir + picloram) highlighted themselves in quickness and efficiency in weed control. The phytointoxication obtained in this treatment was above 84%. which was excellent, since there was only tolerance for

regrowth in only one plant in the average of repetitions. The application of the herbicide with pre-purified glycerin at 5% might be considered as the second most significant treatment to control *A. peregrinum*. However, phytotoxicity scores was below 70% (67.50% at 60 DAA), which was considered a regular checkup, due to the difficulty of controlling such weeds. Plant injury ranges between 50 to 80% can be considered satisfactory. In this treatment, we observed a lower number of sprouts compared to the other treatments, corresponding to a reduction of 54.16% when compared to the control.

According to what was described (Table 4), it can also be observed that at 30 DAA there was no significant difference between treatments that used glycerin plus herbicide (triclopyr + picloram). For the proportion of the applied current commercial standard treatment, which has more diesel and herbicide (triclopyr + picloram) 8% v/v^{-1} , we observed a fast visual phytotoxicity on the plants of *A. peregrinum* differing from all treatments according to the Tukey test at 5% probability. At 60 days after the application, it was observed that the application of glycerine at 5% plus the herbicide (triclopyr + picloram) did not differ statistically from the treatment where diesel oil plus herbicide was used. The diesel acts aggressively on plant tissue, destroying the cuticle and maximizing the efficiency of the herbicide, resulting in a process of senescence. According to Akobundu (1987) and Deuber (1992) the diesel oil presents a phytotoxic effect due to solubilization of cell walls, cell disintegration leading to extravasation and their contents into the intercellular spaces. Among the treatments evaluated (Table 4), in which the glycerin was used in replacement of diesel oil, the use of the herbicide (triclopyr + picloram) more than 5% glycerol (pH 6.20) showed a greater control of *A. peregrinum*. According to Costa et al. (2006) some herbicides have their efficiency increased with a reduction in the pH values of plant close to 4.0. In addition, when it is in lower pH, the hydrolysis rate is delayed, while maintaining a wet sheet for a longer time because the surface of the sheets has a neutral pH, having an interaction with the pH of the syrup. It is important to note that the greater control of *A. peregrinum* was observed only at 60 DAA and may be explained by the physico-chemical properties of the herbicide.

Silva (2005) points out that in general, herbicides that have in the constitution as the active ingredient molecule (picloram) show mainly late effect after application. However they are extremely persistent. Such an inference would be based fast on the metabolization inability of this herbicide by the plant. Therefore the effectiveness of phytotoxicity was a function of exposure time. The lower efficiency of glycerin in replacement of diesel oil can be explained by the aggressiveness capacity of Glycerine on the plant cell wall due to its polar properties, which does not allow an efficient herbicide solubilization and hence a lower penetration in the weed. The stem of *A. peregrinum* contain a significant amount of nitrogen compared to other species and a high lignin

content that makes it the most resistant species in cultivated pastures. The high extractives content present in the *A. peregrinum* stem shows its ability to survive in environments that have extreme conditions. The use of glycerin in replacement of diesel oil to control *A. peregrinum*, was not very efficient when compared to diesel oil. However, it was possible to observe the potential of glycerin as a vehicle for applying herbicides, leaving much to the development of new studies to promote changes in their physico chemical properties.

Conflict of interests

The author(s) did not declare any conflict of interest.

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