

## HERBICIDAL EFFICACY OF ACETIC ACID AND CITRIC ACID BASE ON BROAD LEAF WEEDS OF MEDICINAL CROPS FIELDS

### *Keefektifan herbisida berbasis asam asetat dan asam sitrat terhadap gulma berdaun lebar pada lahan tanaman obat*

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#### ABSTRACT

The study aims to determine the effectiveness of acetic acid based formula on broad leaf weeds grown in medicinal crop fields. Two experiments were conducted, i.e. on small plots (2 m x 3 m) of a farmer's field in Ciapus, Bogor and on a larger plot (2 m x 8 m) at the Cicurug Experimental Station, Sukabumi. The formula consisted of a mixture of acetic acid + NaCl (AG) and acetic acid + citric acid (AC), and formulas of VAC, VACG which were enriched with wood vinegar (V). As control was a commercial synthetic herbicide 2,4 D amine. The acetic acid base formulas applied at two concentrations 10 and 15%, while 2,4-diamine was followed the recommendation (0.3%) using a knapsack sprayer with a flat-fan nozzle tip. Weed vegetations were observed visually before and at 2, 4, 6, and 8 weeks after treatments. Weed severity was recorded using a 0-4 scales (0 = 0-5% mortality ; 1 = 20-50% weeds died ; 3 = 50-75% eradicated and 4 = 75-100% weed were eradicated), as well as re-growth and dry weight of the weeds. The first and second experiment showed that AC, AG, VAC and VACG applied at 10 and 15% were effective in controlling broadleaf weeds. Mechanism of action the formula is a contact poison. Its effectiveness is equivalent to 2,4-D amine treatment dose of 1.5 l ha<sup>-1</sup>. Repeated application is necessary to prolong the effect of herbicide.

**Key words:** Herbicide, acetic acid, citric acid, NaCl, broad leaf weeds

#### ABSTRAK

Penelitian ini bertujuan untuk mengetahui efektivitas formula herbisida berbasis asam asetat pada gulma berdaun lebar yang tumbuh di ladang pada lahan petani di Ciapus, Bogor, dan skala lebih besar (plot berukuran 2 m x 8 m) di Kebun Percobaan Cicurug, Sukabumi. Perlakuan yang diuji adalah campuran asam asetat + NaCl (AG) dan asam asetat + asam sitrat (AC), serta formula yang diperkaya dengan cuka kayu (vinegar; V). Sebagai kontrol adalah herbisida sintesis komersial 2,4 D amina. Konsentrasi herbisida berbasis asam asetat disemprotkan dengan semprotan gendong pada konsentrasi 10 dan 15%, sedangkan 2,4-diamin mengikuti rekomendasi (0,3%). Vegetasi rumput diamati secara visual sebelum dan pada 2, 4, 6, dan 8 minggu setelah perlakuan. Keparahan gulma dicatat dalam skala 0-4 (0 = mortalitas 0-5% ; mortalitas gulma 1 = 20-50% ; 3 = mortalitas gulma 50-75% dan 4 = mortalitas gulma 75-100% , serta pertumbuhan ulang dan berat kering gulma. Hasil percobaan pertama dan kedua menunjukkan bahwa formula AC, AG, VAC, dan VACG yang diaplikasikan pada konsentarsi 10 dan 15% efektif terhadap gulma berdaun lebar. Mekanisme cara kerja formula adalah racun kontak. Efektivitasnya setara dengan 2,4-D pada dosis 1,5 l ha<sup>-1</sup>. Aplikasi berulang diperlukan untuk memperpanjang efek herbisida.

**Kata kunci:** Herbisida, asam asetat, asam sitrat, NaCl, gulma daun lebar

#### INTRODUCTION

Synthetic herbicides are important chemicals in controlling various types of weeds of medicinal crops. The use of herbicides worldwide

is around 49.6% of the total pesticides (Merrington *et al.*, 2002). World pesticide demand has been projected to increase from \$ 26 billion in 2004 to \$ 28.4 billion in the year 2009 with a

growth rate of 1.7% per year (World Pesticide, 2005 in Irianto and Johanis, 2009). The increasing use of herbicides is in line with efforts to meet the global demand for sustainable food, feed and energy (food, feed and fuel). The most widely used of herbicides are glyphosate (N-phosphonomethyl glycine), paraquat (paraquat dichloride), and 2,4-D (2, 4 dichloro phenoxy acetic acid). Although glyphosate is determined as the most least toxic (Duke and Powels, 2008), but recently there has been reports of various negative effects of glyphosate on human healths (FoEE, 2013).

The economic value of herbicides in agriculture is very large, especially in highly intensive agriculture that uses minimal labour. For example, the herbicide glyphosate is very effective in controlling grasses and broadleaf weeds. Weed is often a major problem in crop production systems of food, vegetables, medicinal, and ornamental crops (Hasanuddin *et al.*, 2000). Efforts to get a safer herbicides, such as the use of plant materials and biological, are being actively studied in various countries. However, the results are still limited because of its effectiveness is still far lower than the synthetic herbicides. However, studies showed that synthetic herbicide used can be minimized by mixing with more environmental chemicals, such as wood vinegar, acetic acid or citric acid (Tiilikkala *et al.*, 2010). Wood vinegar (liquid pyrolysis) is the result of condensation of liquid vapor of the combustion process of wood into charcoal at high temperatures (400-500°C) (Tiilikkala *et al.*, 2010). Wood vinegar has long been used in China, Egypt, Greece and India for agricultural purposes, such as fertilizers, pesticides and plant growth stimulants. The main content of liquid smoke is acetic acid and methanol, as well as several other compounds, such as propanoic acid, acetone, methyl acetone, acetaldehyde, allyl alcohol, furan and furfural, and formic, propionic and butyric (Payamara, 2011; Tiilikkala *et al.*, 2010).

Research results of Abouziena *et al.* (2009) showed that acetic acid (5%), citric acid (10%), and clove oil (45.6%) were effective against broadleaf weeds, while the narrow leaf weeds required a higher concentration of acetic acid (30%). Acetic acid is a contact type herbicide and its effect can be seen within hours (1-2 hours after application). Another advantage of acetic acid herbicide is biodegradable, so it does not lead to residues on crops. Controlling weeds with a herbicide mixture of clove oil (318 l ha<sup>-1</sup>) and vinegar (636 l ha<sup>-1</sup>) is quite effective (83%) for controlling weeds in crops of corn, onions and potatoes (Evans and Bellinder, 2009).

An effort to reduce the pressure on the emergence of resistant weeds is to use different types of herbicide alternating or mixing two or more kinds of different types of herbicides. The practice of mixing herbicides divergent types in tanks before being sprayed has been reported, especially to improve the effectiveness, slowing the process of emergence of resistant weeds, reduce herbicide residues, reducing the volume of herbicides and cost required (Damalas, 2004). Rico *et al.* (2007) showed that the mixing of cyhalotop-butyl + bentazone herbicides with wood vinegar (1: 1000) significantly increased the effectiveness of the herbicides while increasing rice yields. Panjehkeh and Alamshahi (2011) showed that the combination of phenmedipham + chloridazon was more effective against broadleaf weeds in sugar beet, without damaging to the leaves and tubers.

This study aims to determine the effectiveness of the mixture of acetic acid + NaCl (AG) and acetic acid+citrate (AC) formulas against several types of broadleaf weeds grown in medicinal crop fields.

## MATERIALS AND METHODS

### Weed identification

Weeds grew on the experimental sites were identified based on their morphological characteristics and their scientific names were

confirmed by using the identification book of weeds in Indonesia.

### First experiment

Acetic acid, NaCl, acid citrate, and commercial herbicide 2,4-D were obtained from local chemical suppliers. The experiment was conducted on a farmer's land at Ciapus, Bogor in 2012. The experiment was a randomized block designed, consisting of six treatments, such as a mixture of acetic acid + NaCl (AG) and acetic acid + acid citrate (AC) with a concentration of 10 and 15%. Used as the formula of commercial herbicides 2,4-D amine 0.3% and the untreated control. Treatment was repeated three times. Treatment plot size was 2 m x 3 m. Spraying is done using a knapsack sprayer with a flat-fan nozzle so that drops of the herbicide solution were evenly spread on the target weeds.

### Second experiment

This experiment was conducted at the Cicurug Experimental Station, Sukabumi. Field conditions were evenly grown with natural weeds. Plot size was 2 m x 8 m. Thirteen treatments were evaluated, such as (1) Formula AG 15%, (2) Formula AG 20%, (3) Formula AC 15%, (4) Formula AC 20%, (5) Formula ACG 15%, (6) Formula ACG 20%, (7) Formula VACG 15%, (8) Formula VACG 20%, (9) Glyphosate, (10) Paraquat, (11) 2,4-D amine, (12) mechanical daytime, and (13) Control. The treatment was designed as a randomized block design (RBD), repeated three times.

Application of the formula AG, AC and ACG performed three times with interval one week, while the commercial herbicide was applied once. Manual weeding was done during twice with a three weeks interval.

Parameters measured were (a) the percentage of the target weed mortality, (b) weed re-growth, and (c) dry weight of cut weeds.

### Parameter observation

Prior to application of herbicide, weed vegetation was observed visually to determine the

types of dominant weeds. After the application, parameters measured were (a) weed mortality of 0-4 scales observed in 2, 4, 6, and 8 weeks after application (MSA), (b) percentage of weed re-growth, and (c) new weed emergence. The weed mortality rate was calculated as follows. Scale 0 means up to 0-5% mortality; scale 1 (lowest category) means 5-20% weed died; scale 2 (mild category) means 20-50% weeds died; scale 3 (medium category) means 50-75% eradicated; and scale 4 (severe category) means 75-100% weed were eradicated. (Komisi Pesticida, 2000; Pujiswanto, 2011).

The effectiveness of herbicide formula is calculated by comparing the mortality rates of weeds in plots treated AG and AC against weed mortality in the control plots.

## RESULTS AND DISCUSSION

### Weed identification

In the first experiment at farmer's field in Ciapus, Bogor, various broad leaf weeds identified were *Ageratum conyzoides*, *Synedrella nodiflora*, *Borreria alata*, *B. laevis*, *Phyllanthus niruri*, *Euphorbia hirta*, *Mimosa Invisa*, *Erechtites valerianifolia* (*Crassocephalum crepidioides*), *Sida rhombifolia*, *Amaranthus dubius* (*spinach spines*), *Diodia sarmentosa*, and *Galinsoga parviflora*, whereas narrow weeds were *Digitaria ciliaris*, *Imperata cylindrica*, *Eleusine indica*, *Axonopus compressus*, *Cyperus rotundus* and *C. kyllingia*. All the weeds found the experimental plots, although the most dominant was *A. conyzoides* and *B. alata*, whereas *P. niruri* and *E. Hirta* were growing unequal in the plots.

In the second experiment at the Cicurug Experimental Station, Sukabumi, 17 weeds were identified, but the most dominant were *Ageratum conyzoides*, *Borreria latifolia* and *Cynedrella nodiflora* (broad leaf weeds), and *Digitaria ciliaris* (narrow leaf weeds), such as *Cyperus rotundus*.

### First experiment

Weeds sprayed with the formula of acetic

acid (AC and AG treatment) instantly dried up like burning leaves within 1-2 hours after the treatment. Broad leaf weeds, such as *B. alata*, *A. conyzoides*, *S. nodiflora*, *P. niruri* and *E. hirta* were the most sensitive. According Tjokrowardojo and Djauhariya (2011) those weeds (*Ageratum conyzoides*, *Synedrella nodiflora*, *Borreria alata*, *Borreria laevis*, *Axonopus compressus*, *Cynodon dactylon*, *Digitaria ciliaris*, *Eleusine indica*, *Cyperus rotundus* and *Cyperus kyllingia* were dominant in ginger cultivating areas and caused a significant yield reduction.

One day after the treatment, more obvious burning symptoms were observed in all plots. This suggests that this acetic acid base herbicide acts as a contact poison. Similarly, Abouziena *et al.* (2009) and Evans and Bellinder (2009) found that 30% acetic acid and 10% citric acid solutions were effective against broad leaf weeds. However, the narrow leaf weeds such as grass did not affect. Two weeks after the application, all weeds were completely dried (scores 3-4) in all treated plots. The higher the concentration the more severe the symptoms (Table 1). Effectiveness of the two formulas was

lasting from three weeks after the application. Its effectiveness is equivalent to 2,4-D amine rate at 1.5 l ha<sup>-1</sup>.

Treatment formula AG (10 and 15%) which contains acetic acid and salts, as well as air conditioning formula that contains acetic acid + citrate acid (10 and 15%) growth does not occur again (regrowth) after one month of application; means both treatment deadly weed perfectly. However, on a plot that had been treated with the second formula, three weeks after treatment grow other types of weeds (Table 2). Percentage weed cover just reached less than five percent in the Acetic acid Citric acid (AC) and Acetic acid + salt NaCl (AG) treatment, and 2,4D, whereas in the control treatment weeding manually (hand weeding) percentage reaches 20-30%, meaning that most of the experimental plot was overgrown with weeds. The low percentage of weed cover, although in the control treatment without treatment, is also related to the weather conditions are getting dry (dry season), where the growth of new weeds getting smaller. This reflects that the herbicide AC and AG are a growing herbicide post (post-emergence) and contacts that are not deadly weed seeds in the tillage layer.

Table 1. Severity of acetic acid and citric acid base formulas on target weeds, three weeks after treatment.

Tabel 1. Keparahan pada gulma target setelah aplikasi formula berbasis asam asetat dan asam sitrat, tiga minggu setelah aplikasi.

Treatment <sup>a)</sup>	Severity (%) <sup>b)</sup>				
	<i>Borreria alata</i>	<i>Ageratum conyzoides</i>	<i>Synedrella nodiflora</i>	<i>Phyllanthus niruri</i>	<i>Euphorbia hirta</i>
AG 10	3	3	3	4	4
AG15	3	4	4	4	4
AC10	3	4	3	4	4
AC15	4	3	4	4	4
2,4D 0,3	3	4	4	4	4
Control	0	0	0	0	0

Note: <sup>a)</sup> AG = acetic acid + NaCl; AC = acetic acid + citric acid; 2,4 D = 2, 4 dichloro phenoxy acetic acid.

<sup>b)</sup> Severity score: 0 = no effect or very low (0-5% toxicity), 4 = severe (75-100% toxicity).

Keterangan: <sup>a)</sup> AG= asam asetat + NaCl; AC= asam asetat + asam sitrat; 2,4D dichloro phenoxy acetic acid.

<sup>b)</sup> Nilai keparahan: 0=tidak ada efek atau rendah keracunannya (0-5% toksisitas), 4=sangat beracun (75-100% keracunannya).

## Second experiment

The effect of spraying of herbicide solutions on target weeds was presented in Table 3. The result showed the acetic acid base formula (acetic acid + NaCl and acetic acid + citric acid) applied at 15 and 20% was as effective as synthetic herbicide 2,4 D sprayed at the recommended dosage (0.3%). The combination of the three substances, i.e. acetic acid + citric acid + NaCl did not significantly differ from that of the two combinations. The effect of wood vinegar in the formula of VACG did not increase its effectiveness, but the addition of wood vinegar is important for reducing aroma of acetic acid.

Incorporating of NaCl in the formula may not be necessary, because NaCl is not degradable in the soil.

## Weed recovery

The results showed that before application, the land was mainly dominated with broad leaves, such as *Ageratum conyzoides*, *Borreria latifoli* and *Cynedrela nodiflora*. All the acetic acid base formulas tested (AC, AG, ACG and VACG) were toxic to broad leaf weeds shown as leaf burn (score three out of four) one hour after application. Six weeks following application, dry weight of the weeds remained less than less than five percent (none to few broad leaf weed

Table 2. Regrowth of weeds three weeks after treatment.

Table 2. *Pertumbuhan kembali gulma tiga minggu setelah aplikasi.*

Treatments <sup>a)</sup>	Weed coverage (%) <sup>b)</sup>				
	<i>Borreria alata</i>	<i>Ageratum conyzoides</i>	<i>Synedrella nodiflora</i>	<i>Phyllanthus niruri</i>	<i>Euphorbia hirta</i>
AG 10	5	5	5	5	5
AG15	5	5	5	5	5
AC10	5	5	5	5	5
AC15	5	5	5	5	5
2,4D (0.3)	5	5	5	5	5
Control	30	35	20	10	5

Note: <sup>a)</sup> AG = acetic acid+ NaCl; AC = acetic acid + citric acid; 2,4 D = 2, 4 *dichloro phenoxy acetic acid*.

<sup>b)</sup> Severity score: 0 = no effect or very low (0-5 % toxicity), 4 = severe (75-100 % toxicity).

Keterangan: <sup>a)</sup> AG= asam asetat + NaCl; AC= asam asetat + asam sitrat; 2,4D *dichloro phenoxy acetic acid*.

<sup>b)</sup> Nilai keparahan: 0=tidak ada efek atau rendah keracunannya (0-5% toksisitas), 4=sangat beracun (75-100% keracunannya)

Table 3. Severity of acetic acid base herbicide formulas on several weeds grown in the Cicurug Experimental Station.

Table 3. *Keparahan pada beberapa jenis gulma setelah aplikasi formula berbasis asam asetat dan asam citrat, tiga minggu setelah aplikasi.*

Treatment <sup>a)</sup>	Severity scores of 0-4 scales (%)						
	<i>Ageratum conyzoides</i>	<i>Borreria latifolia</i>	<i>Cynedrela nodiflora</i>	<i>Cleome aspera</i>	<i>Amaranthus sp</i>	<i>Mimosa invisa</i>	<i>Mimosa pudica</i>
AC (15)	4	3	4	4	3	3	3
AC (20)	4	3	4	4	3	3	3
AG (15)	4	3	4	4	4	3	3
AG (20)	4	4	4	4	3	4	3
ACG (15)	4	4	4	4	4	3	3
ACG (20)	4	4	4	4	4	4	3
VACG (15)	4	4	4	4	4	4	4
VACG (20)	4	4	4	4	4	4	4
2.4-D (0.3)	4	4	4	4	4	4	4
Control	0	0	0	0	0	0	0

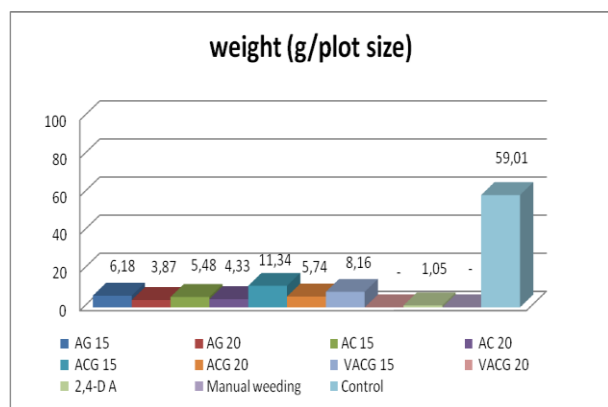
Note: <sup>a)</sup> AC = acetic acid + citric acid; AG = acetic acid + NaCl; ACG = acetic acid + citric acid+NaCl; VACG = wood vinegar + acetic acid + citric acid + NaCl.

<sup>b)</sup> Severity score: 0-5 scales; 0 = 0-5 % toxicity, 4 = 75-100% toxicity.

Keterangan: <sup>a)</sup> AG= asam asetat + NaCl; AC= asam asetat + asam sitrat; 2,4D *dichloro phenoxy acetic acid*.

<sup>b)</sup> Nilai keparahan: 0=tidak ada efek atau rendah keracunannya (0-5% toksisitas), 4=sangat beracun (75-100% keracunannya).

regrowth on plot experiments) compared with the standard herbicide treatment (Figure 1). However, there was a shift domination of grass weeds on the plots. Acetic acid content in the formulas ranged from 1-30%, depending on the formulas.



Note/Keterangan: AC = acetic acid+ citric acid; AG = acetic acid + NaCl; ACG = acetic acid + citric + NaCl; VACG = wood vinegar + acetic acid + citric + NaCl.

Figure 1. Weed dry weight on experimental plots treated with acetic acid and citric base formula in the Cicurug Experimental Station.

Gambar 1. Berat kering gulma pada plot percobaan yang diperlakukan dengan formula berbasis asam asetat dan asam sitrat di KP. Cicurug.

The results showed that the herbicide formula contains a mixture of acetic acid and acetic acid + NaCl + citric acid potential as herbicides, especially for the types of broadleaf weeds, such as *B. alata*, *A. conyzoides*, *S. nodiflora*, *P. niruri* and *E. hirta*. However, the main constraint is the amount of concentration that is still too much (10-15%) making it less able to compete with existing commercial formulas that use the average concentration of 0.5-1%. To reduce the magnitude of the effective concentration of the herbicide formula AG or AC it is necessary to study the effect of a mixture of formula herbicide AG or AC with herbicide other active ingredients. Acenas *et al.* (2013) showed that a mixture of liquid smoke (pyroligneous acids) containing the main compound with acetic acid herbicide butyl bentazone + cyhalof more effective to control broad leaf weeds. The main drawback of the formula mixture of acetic acid

and acetic acid + NaCl + citric acid is a very pungent odour. To that end, the improvement of the formula needs to be done to reduce the odour, such as by incorporating liquid smoke in the formulation. Liquid smoke can neutralize the scent, (Rico *et al.*, 2007; Evans and Bellinder 2009) it can be used as a solvent in the formula herbicide mixtures containing oil of cloves and active ingredients of other herbicides. These herbicides can be applied to the cultivation of medicinal plants of the family of the gramineae.

## CONCLUSION

An acetic acid base formula containing acetic acid, NaCl, citric acid and wood vinegar applied at 10-15% were effective in controlling broadleaf weeds on medicinal crops's land such as *B.alata*, *A. conyzoides*, *S. nodiflora*, *P. niruri* and *E. hirta*. Mechanism of action of the herbicide formula is a with contact poison. The most promising formula is acetic acid + citric acid + wood vinegar. However, the formula did not affect grasses.

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