International Journal of Science and Research (IJSR)
ISSN: 2319-7064
ResearchGate Impact Factor (2018): 0.28 | SJIF (2018): 7.426

Herbicidal Effect of Concentrated Acetic Acid Alone and its Clove Polar Compounds Extract Formulation on Mono and Dicotyledonous Weeds

Eskander M. A[1], H. I. Abd-Alla[2], Farag E. M.[3]

Pesticide Formulations Research Department, Central Agricultural Pesticides Lab. (CAPL), Agricultural Research Center (ARC), Dokki, Giza, Egypt

Abstract: ***Polar compounds of clove were extracted with acetic acid as a solvent, the crude extract was qualitative phytochemically screened, and saponins, tannins and traces of alkaloids were detected in it. The crude extract with solvent and the solvent were tested on wheat and cucumber as patterns for mono and dicotyledonous plants under laboratory conditions. The crude extract showed high inhibition effect on germination, root and shoot growth of both plants under study although the effect was greater on wheat than cucumber. In addition, the effect of the crude with acetic acid was higher than that of acetic acid used alone. The crude was then formulated as 10 % soluble concentrate formulation (SL). The new formula succeeded to pass all tests specified for soluble concentrates. It was then tested on wheat and cucumber under laboratory conditions. The new formula also inhibited germination, root and shoot growth of both plants by greater values compared to that of the crude with solvent (a.i). Under greenhouse conditions, the new formula and the solvent were tested as post emergence treatment on Phalaris minor and Rumix dentatus as patterns for mono and dicotyledonous weeds as a foliar application. The results showed clearly that formulated clove extract 10%SL at 4% gave 95% burning on phalaris minor and 80% on Rumix dentatus, however acetic acid (solvent) at 4% gave 65% on phalaris minor and 60% Rumix dentatus after 14 days of application. Also the formulated extract showed greater effect as reduction of shoots fresh and dry weight than acetic acid (solvent) alone. The effect that appeared as burning and reduction in shoots fresh and dry weight but the effect on monocotyledons was greater than that on dicotyledons.***

Keywords: Clove, Formulation and Post-emergence Herbicides

1. Introduction

Weeds are plants in the wrong place, it affect everyone in the world by reducing crop yield and crop quality, delaying or interfering with harvesting, interfering with animal feeding (including poisoning), reducing animal health, preventing water flow, as plant parasites, etc. (**Abouziena and Haggag, 2016**). **Rao** (**2000**) has reported that of the total annual loss of agricultural produce from various pests, weeds account for 45 %, insects 30 %, diseases 20 % and other pests 5 %. Annual worldwide losses to weeds were estimated to comprise approximately 10-15 % of attainable production among the principal food sources.

Chemical weed control began just about a century ago with a few inorganic compounds, such as sulfuric acid, copper salts, and sodium chlorate (**Cremlyn, 1991**). The herbicidal activity of 2, 4-dichlorophenoxyacetic acid was detected in the 1940s (**Troyer, 2001**). Environmental pollution and contamination of surface water and groundwater by herbicides is one of the most important human concerns (**Abdin *et al.*, 2000**), in addition herbicide resistance in weeds has developed into a more serious problem that now constrains the application of certain types of herbicides in some markets (**Kraehmer *et al.*, 2014**). Thus, there is an importance to search for environmentally safer and novel compounds with more effective, more specific targets for the management of weeds. Recently, many studies have investigated the phytotoxic potentials of plant extracts and individual compounds and their ability to control weeds in crop production (**Choi *et al.*, 2015**).

Allelochemical from *Vicia villosa* Roth, where cyanamide had a phytotoxic effect on onion root growth by disturbance of cell division and by inhibition of proliferation of meristematic cells and cell cycle. **Soltys *et al*. (2011)** also reported that, the effects of phytotoxins on plants are diverse the phytotoxic activity of allelochemicals usually involves alteration in one of the crucial physiological process such as photosynthesis, respiration, induction of ROS and cell division, which all lead to cell death. Inhibition of any of these processes results in disturbances in plant growth and development.

The main constituent of clove plant is clove oil; it may be derived from flower buds, leaves and stems. The principal biologically active component is eugenol. Clove oil exhibits phytotoxicity, which means it has herbicidal activity; Clove oil is applied as post-emergent, non-selective, and broad-spectrum. Clove oil's disruption of cell membranes took place under both dark and light conditions **(Baker *et al.*, 2018).**

The present study was undertaken to evaluate the herbicidal activity of crude containing polar materials (saponins, tannins and alkaloids) extracted from clove (*Syzygium aromaticum*) flower buds in acetic acid as natural alternatives of herbicide and formulate the filtrate crude extract in the form of soluble concentrate for use in the field of weed control.

2. Materials and Methods

2.1. Tested chemicals

a) Acetic acid (ethanoic acid, molar mass 60.05 g.mol^{-1}), was supplied by EL-Gomhoria Co., Cairo, Egypt.

b) Clove (Syzygium aromaticum), was supplied by Agricultural Development Markets, Nadi Alsayd, Dokki, Giza, Cairo, Egypt.
c) Surface active agents: Tween 20, Tween 40 and Tween 80 were supplied by EL-Gomhoria Co., Cairo, Egypt.

2.2. The physico-chemical properties of basic formulation constituents:

2.2.1. Active ingredient

a) Solubility: It was determined by measuring the volume of distilled water, acetone and xylene for complete solubility or miscibility of one gram of active ingredient at 20 ˚C **(Nelson and Fiero, 1954)**. The % Solubility was calculated according to the following equation:

% solubility = W/V x 100

[Where; W= active ingredient weight, V= volume of solvent required for complete solubility].

b) Free acidity or alkalinity: It was determined according to the method described by **WHO specification** (**1979**).

2.2.2. Surface active agents

a) Surface tension: It was determined by using Du-Nouy tensiometer for solutions containing 0.5 % (W/V) surfactant according to **ASTM D-1331** (**2001**).
b) Critical micelle concentration (CMC): The concentration in which the surface tension of solution doesn't decrease with further increase in surfactant concentration, (CMC) of the tested surfactants was determined according to the method described by (**Osipow, 1964**).
c) Hydrophilic-lipophilic balance (HLB): The solubility of surfactant in water is considered as approximate guide to its hydrophilic-lipophilic balance (**Lynch and Griffin, 1974**).
d) Free acidity or alkalinity: It was determined by the same method described before.

2.2.3. Preparation of soluble concentrate formulation

Crude extract of clove was prepared as 10 % SL using different wetting agents added to clove extract by serial concentrations, and then the physico-chemical properties were measured. Depending on surface tension for the prepared formulas the best one was chosen to complete other formulation and bioassay testes.

a) Free acidity or alkalinity: It was measured as mentioned before.

b) Surface tension: It was determined as mentioned before.

2.2.4. The physico- chemical properties of spray solution at field dilution rate (0.5 %).

a) Surface tension: It was determined as mentioned before.
b) pH: It was determined by using Cole-Parmer PH conductivity meter 1484-44 according to **Dobrat and Martijn** (**1995**).
c) Viscosity: It was determined by using Brookfield viscometer Model DVII+Pro, where centipoise is the unit of measurement according to **ASTM D-2196** (**2005**).
d) Electrical Conductivity: It was determined by using Cole-Parmer pH/Conductivity meter 1484-44, where µmhos is the unit of electrical conductivity measurements according to **Dobrat and Martijn** (**1995**).

2.3 Extraction Method

Clove powder was extracted in acetic acid 36 % as a solvent according to (**Handa *et al.*, 2008**).as described below:

100 grams of clove powder was macerated in 500 ml acetic acid in a stoppered container for three days at room temperature with frequent agitation, and then the mixture is pressed or strained by filtration. In this conventional method the choice of solvents will determine the type of compound extracted from the samples.

2.4 Qualitative phytochemical analysis of clove crude extracted with acetic acid

Preliminary phytochemical analysis was carried out for the extract as per standard methods described by **(Santhi and Sengottuvel 2016)**

2.5 Bioassay

2.5.1 Under laboratory conditions

Inhibition effect of acetic acid 36 %, clove crude extract and its 10 % soluble concentrate (SL) formulation on seed germination root and shoot growth was carried out according to the procedure described by **Powel and Spencer** (**1988**) with some modifications as described below:

Serial concentrations from acetic acid 36 %, clove crude extract and its soluble concentrate (SL) formulation were prepared by dissolution in water. The calculated amount from each concentration was pipette on thirty seeds of wheat and cucumber as patterns for mono and dicotyledonous plants and agitated to coat the seed surface. Each ten seeds were transferred to Petri dish (90 mm diameter), lined with a dry filter paper, 6 ml distilled water were pipette on the filter paper, Petri dish was then sealed with (PVC) electrical insulating tape. After complete germination of control (Petri dishes containing untreated seeds), the number of germinated and non-germinated seeds and radical roots and shoots length were recorded. Three replicates were done for each treatment **(El-kady *et al*., 2000).**

2.5.2 Under greenhouse conditions (Foliar application):

The efficacy of the formulated clove extract 10 % SL at the concentration of 4, 2, 1, 0.5 % and acetic acid 36 % at the concentration of 4 % were assessed against *Phalaris minor* Retz. as pattern for monocotyledonous weeds and *Rumex dentatus* as pattern for dicotyledonous weeds as follow: Three plastic pots for each concentration were filled till their lower surface by mixture of peat moss and sand (1:3).while the weed seeds difficult to count so 0.25 gr seeds of *Phalaris minor* Retz and 0.5gr seeds of *Rumex dentatus* were planted in each experimental pot and irrigated, after 15 days from planting sprayed by the calculated concentration of spray solution of the tested materials , irrigated with water according to need, then compared with untreated pots taken as control for post-emergence treatment, **(Hussein, 1989).**

2.6 Statistical analysis

Inhibition percentages were corrected by using **Abbott's formula** (**1925**), and the concentration inhibition regression lines were drawn according to the method of **Finney** (**1952**).

3. Results and Discussion

A soluble concentrate (SL) is a clear solution to be applied as a solution after dilution in water. Soluble concentrates are based on either water or a solvent mixture which is completely miscible in water. Solution concentrates are the simplest of all formulation types and merely require dilution into water in the spray tank (**Zabkiewicz, 2000**).

The physico-chemical properties of clove extract (a.i) were studied to determine which kind of formulation will be suitable, in addition, that of the surface active agents used.

3.1. Physico-chemical properties of clove extract as active ingredient

Clove extract showed complete solubility in water, miscible in acetone and complete immiscibility in xylene. It showed a high degree of acidity obtained from the value calculated as sulfuric acid percentage (11.36), this high value of acidity is due to the extract contained also acetic acid. These results direct the processes of formulation to solutions (soluble concentrates, liquids) as the active ingredient showed complete solubility in water (100 %) Table (1).

Table 1: Physico-chemical properties of clove extract as active ingredient.

Solubility % (W/V)			Free acidity as % H_2SO_4
Water	Acetone	Xylene	
100	miscible	Non miscible	11.36

3.2. Physico-chemical properties of surface active agents

Table (2) showed the physico-chemical properties of three surfactants; Tween 20, 40 and 80. All of them showed relative surface tension values 36, 37 and 39 dyne/cm for the three respectively mentioned surfactants. Also all of them showed HLB value greater than 13 this mean that all of these surfactants soluble in water and suitable for preparation the extract as soluble liquid formulation. But the three surfactants showed different CMC %; 0.2, 0.4 and 0.5 for the three formerly mentioned surfactants respectively. Although the three surfactants were acidic, Tween 80 showed the highest value (0.50) followed by Tween 40 (0.13) and Tween 20 (0.02). These results revealed that any of the three surfactants could be used in formulating clove extract as soluble concentrate (SL).

Table 2: Physico-chemical properties of surface active agents

Surface active agent	Surface tension dyne/cm	HLB	CMC %	Free acidity as % H_2SO_4
Tween 20	36	>13	0.2	0.02
Tween 40	37.02	>13	0.4	0.13
Tween 80	39.2	>13	0.5	0.50

3.3. Physico-chemical properties of the local prepared clove extract as 10 % soluble concentrate formulation before and after accelerated storage:

The physico-chemical properties of the new formula were determined under normal storage conditions and after accelerated storage conditions, results obtained in Table (3)indicated that most of the tested physical properties showed no changes under both storage conditions (solubility, sedimentation and appearance). Free alkalinity changed after accelerated storage compared with before storage from 7.20 - 8.16, the same result was obtained with surface tension as it was increased after accelerated storage compared with before storage from 37.60 to 38.60 dyne/cm.

Table 3: Physico-chemical properties of the 10 % soluble concentrate local formulation before and after accelerated storage

Before storage					After storage				
Surface tension dyne/cm	Free acidity as % H_2SO_4	Solubility	Sed*.	App**.	Surface tension dyne/cm	Free acidity as % H_2SO_4	Solubility	Sed*.	App**.
37.6	7.2	soluble	Nil	clear	38.6	8.16	soluble	Nil	clear

Sed*.: Sedimentation App**.: Appearance

3.4. Physico-chemical properties of spray solution at field dilution rate (0.5 %)

The physical properties of the spray solution are closely related to the biological activity of the new formula Table (4). The spray solution showed high viscosity 1.037 centipoise, the increase in viscosity was expected to increase the pesticidal efficiency according to (**Spanoghe *et al.*, 2007)** as it causes reduction drift and retention sticking. Also it showed high electrical conductivity and low pH values (acidic , 5.15), the increase in electrical conductivity and the decrease in pH value of the spray solution would led to deionization of insecticide, increase its deposits and penetration in the treated surface with a consequence increase in its insecticidal efficacy as stated by **Twfik and El-Sisi, 1987**. On contrast to viscosity and electrical conductivity, the decrease in surface tension compared to that of water (39.2 dyne/cm to 72 dyne/cm) could result in a significant increase in the pesticidal activity, **Ryckaert *et al.*, 2007** reported that, the decrease in surface tension could increase spreading on the treated surface with a consequence increase in pesticidal efficacy.

Table 4: Physico-chemical properties of spray solution at field dilution rate (0.5 %)

Viscosity centipoise	Electrical conductivity µ mhos	pH	Surface tension dyne/cm
1.037	552	5.15	39.2

Phytochemical part

Phytochemicals are the chemical produces by various parts of the plants. Theses bioactive constituents of plants are steroids, terpenoides, carotenoids, flavonoids, alkaloids, tannins and glycosides. These compounds have various activities such as antimicrobial and antibacterial, some have been reported to exhibit hemolytic and foaming activity reported by **Feroz *et al*. (1993).**

Qualitative phytochemical screening will help to understand a variety of chemical compounds produced by plants and quantification of those metabolites will help to extract, purify and identify the bioactive compounds for useful aspects to human being **Santhi and Sengottuvel, (2016).**

The present study was carried out on clove (*Syzygium aromaticum*) flower buds revealed the presence of active phytochemical compounds. Qualitative analysis of these compounds in crude extract revealed the presence of saponins, tannins and traces of alkaloids Table (4).

Table 4: Qualitative phytochemical analysis of *(Syzygium aromaticum)* extracted with acetic acid as a solvent

No	Phytochemicals	Acetic acid extracts (flower buds)
1	Saponins	+ + +
2	Tannins (pyrogalol)	+ +
3	Alkaloids	+ (traces)
4	Flavonoids	_
5	Phenols	_
6	Steroids	_
7	Terpenoids	_

Saponins are secondary metabolites synthesized by many different plant species and marine animals. They are large molecules and contain a hydrophobic part, composed of triterpenoid (30 carbon atoms) or steroid backbone (27 carbon atoms with a 6- ring spirostone or a 5- ring furostane skeleton) and a hydrophilic part consisting of several saccharide residues linked to the hydrophobic scaffold through glucose bonds. They have many medical uses including microbial, anti- tumor, anti-insect hepatoprotective, haemolytic and anti-inflammatory activities. **Barbosa, (2014)**

So saponins have many modes of actions against different living organisms like cytotoxic and antitumor activity, also **Glauert, *et al*. (1962**) proposed that the first model for the mechanism of action of saponins cause an increase in membrane permeability.

3.5 Bioassay results

3.5.1 Under laboratory conditions:

Acetic acid 36 %

Acetic acid was used as solvent to extract polar materials from clove to be used as alternatives for chemical herbicide; its herbicidal efficacy was assessed under laboratory conditions (petri- dishes). Table (6) showed that, acetic acid inhibited wheat seeds germination, roots and shoots growth and the EC_{50} values were 86.06, 44.93 and 53.16 ppm respectively. Acetic acid was more effective on roots and shoots growth than the seed germination.

Table 6: Effect of acetic acid 36 % on germination, root and shoot growth of wheat as pattern for monocotyledonous plants under laboratory conditions

Concentration	% of inhibition of		
ppm	Germination	Root growth	Shoot growth
10	0	4.185	2.44
20	0	17.55	12.37
40	5.28	44.68	36.83
80	43.87	74.68	68.57
160	90.5	92.82	90.37
Slope	4.87	2.65	2.72
EC_{50}	86.06	44.93	53.16
EC_{90}	157.77	136.65	157.0

Data in Table (7) showed the efficacy of acetic acid on cucumber seeds germination, roots and shoots growth as pattern of dicotyledonous plants, and it inhibited strongly the three tested parameters, their EC_{50} values were 92.12, 42.44 and 47.22 ppm respectively. The efficacy was increased by increasing concentration. From Table (6 & 7) it was found that, monocotyledonous plants(wheat) were more sensitive to acetic acid in the germination than dicotyledonous plants(cucumber), in contrast, with roots and shoots growth, dicotyledonous plants were more sensitive than monocotyledonous to acetic acid under laboratory conditions.

Table 7: Effect of acetic acid 36 % on germination, root and shoot growth of cucumber as pattern for dicotyledonous plants under laboratory conditions

Concentration	% of inhibition of		
ppm	Germination	Root growth	Shoot growth
10	0	4.05	4.29
20	0	18.18	17.1
40	1.48	47.17	42.7
80	35.49	77.78	72.03
160	92.64	94.52	91.16
Slope	6.1	2.78	2.55
EC_{50}	92.12	42.44	47.22
EC_{90}	150.02	122.72	150.21

1) Crude extract of clove

Clove extract was tested under laboratory conditions on wheat as pattern for monocotyledonous plants by serial concentrations on three plant parameters seed germination, root and shoot growth Table (8). The extract strongly inhibited germination, root and shoot growth, which was evident from the inhibition percentages; in addition, a gradual direct relation between concentration and the percentage of inhibition was found for the three parameters. Slope values for root and shoot were very close indicating the same mode of action, EC_{50} values also referred to high efficacy of the extract on both parameters of wheat compared to germination, but the effect was higher on root than in shoot and germination.

Table 8: Effect of clove extract on germination, root and shoot growth of wheat as pattern for monocotyledonous plants under laboratory conditions

Concentration ppm	% of inhibition of		
	Germination	Root growth	Shoot growth
10	0	9.82	7.11
20	1.53	28.04	22.32
40	14.54	55.14	47.81
80	52.76	79.95	74.26
160	88.39	93.95	91.26
Slope	3.74	2.36	2.34
EC_{50}	76.65	35.26	42.21
EC_{90}	168.72	123.04	148.45

Table (9) showed the effect of the extract on cucumber as pattern for dicotyledonous plants on the same parameters previously mentioned in case of wheat under laboratory conditions. The extract showed the same effect on cucumber in the same way as it affected the monocotyledons and also the effect was higher on the root compared to that of shoot and germination; this is evident from the EC_{50} values as it was lower in case of the root (30.90 ppm) than that in case of the shoot (35 ppm) and germination (82 ppm).

Table 9: Effect of clove extract on germination, root and shoot growth of cucumber as pattern for dicotyledonous plants under laboratory conditions

Concentration ppm	% of inhibition of		
	Germination	Root growth	Shoot growth
10	0	14.92	11.25
20	0	34.42	29.39
40	8.35	59.4	55.16
80	48.11	80.96	78.85
160	90.09	93.49	92.96
Slope	4.43	2.12	2.23
EC_{50}	81.99	30.90	35
EC_{90}	159.51	124.15	131.31

The results obtained from the testing of the extract on mono and di cotyledons Tables (8 and 9) encouraged the processes of extract formulation in a local commercial form.

2) Clove extract 10 % soluble concentrate formulation

The efficiency of the new formula on wheat under laboratory conditions by serial concentrations represented in Table (10). It showed high inhibition effect on the three parameters under study, as there were direct gradual uniform increase in the percentage of inhibition and concentration for all parameters.

Table 10: Effect of clove extract 10 % soluble concentrate formulation on germination, root and shoot growth of wheat as pattern for monocotyledonous plants under laboratory conditions

Concentration ppm	% of inhibition of		
	Germination	Root growth	Shoot growth
10	0	16.41	13.52
20	4.22	38.72	32.1
40	22.59	65.74	56.74
80	58.79	86.36	78.97
160	88.42	96.29	92.51
Slope	3.23	2.29	2.11
EC_{50}	68.31	26.65	33.24
EC_{90}	169.94	96.26	134.40

Clove extract 10 % soluble concentrate formulation inhibited germination, root and shoot growth of cucumber as pattern for dicotyledonous plants under laboratory conditions by the same way as in case of wheat. Also a gradual proportionating relation was found between the increase in concentration and the increase in the percentage of inhibition for all parameters under study Table (11). From Tables 8, 9, 10 and 11 and the EC_{50} values the clove extract and its 10 % SL formulation were more effective on wheat seeds germination than cucumber seeds. While the root and shoot growth of cucumber were more sensitive than that of wheat , and on comparing root and shoot growth of cucumber, root growth were more affected than shoot growth.

Table 11: Effect of clove extract 10 % soluble concentrate formulation on germination, root and shoot growth of cucumber as pattern for dicotyledonous plants under laboratory conditions

Concentration ppm	% of inhibition of		
	Germination	Root growth	Shoot growth
10	0	22.18	16.96
20	1.68	45.91	38.08
40	15.44	71.26	63.66
80	54.14	88.96	84.18
160	88.97	97.03	95.09
Slope	3.72	2.20	2.16
EC_{50}	75.02	22.26	27.60
EC_{90}	165.64	84.93	107.65

Table (12) showed comparison between the efficiency of clove extract 10 % SL and acetic acid 36 % on germination, root and shoot growth of wheat and cucumber under laboratory conditions. The polar materials in clove extract adding and increased the herbicidal activity of acetic acid it showed clear in decreased the EC_{50} values of wheat and cucumber germination, roots and shoots growth, it means increase the inhibition effect by low concentration of the clove extract 10% SL which contain acetic acid as solvent for extraction the polar materials (saponins, tannins, and alkaloids) and solvent don't evaporate but used all the filtrate crude extract to formulate 10% SL. It means the clove extract 10 % SL has both mode of action for acetic acid and extracted phytochemicals.

Table 12: Increase in effectiveness of clove extract 10 % SL formulation compared to acetic acid on germination, root and shoot growth of wheat and cucumber under laboratory conditions

Parameter / Compound	Wheat parameters EC_{50} (ppm)			Cucumber parameters EC_{50} (ppm)		
	Germination	Root growth	Shoot growth	Germination	Root growth	Shoot growth
Acetic acid 36%	86.06	44.93	53.16	92.12	42.44	47.22
Clove extract 10%SL	68.31	26.65	33.24	75.02	22.26	27.6

a- Under greenhouse conditions

Serial concentrations from clove extract 10 % SL and acetic acid 36 % were used to treat *Phalaris minor* and *Rumix dentatus* weeds under greenhouse conditions as post-emergence treatment (foliar application after 14 days of sowing)

Figure 1: Extract 4000ppm control day 10

Figure 2: acetic acid 4 % control day 10

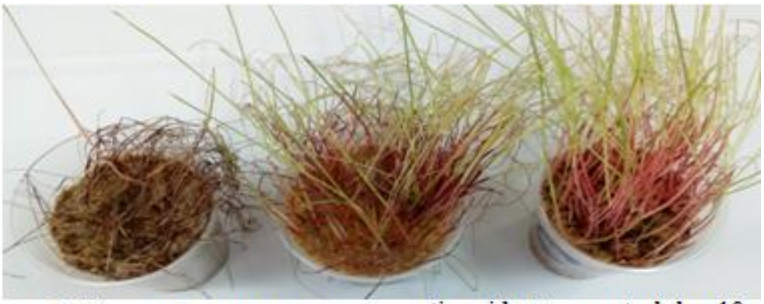

Figure 3: Clove extract 4000 ppm acetic acid 4% control day 10

Figure 4: Clove extract 4000 ppm acetic acid 4% control day 10

Data in table (13) and figures 1, 2 and 3 showed the efficacy of clove extract 10 % SL and acetic acid against *Phalaris minor* as pattern of monocotyledonous weeds under greenhouse conditions, where clove extract at 4 % gave the best efficacy appeared as burning and reduction in shoots fresh and dry weight and the efficacy values were 95, 65.2, and 50.5 % respectively. However acetic acid at 4 % showed low efficacy against the tested weed, the percentage of efficiency burning and reduction in shoots fresh and dry weight were 65, 17.32 and 25.54 % after 14 days from application respectively.

The obtained results were consistent with **Abouziena *et al.* (2009)** as they reported that acetic acid (5 %) and clove oil (45.6 %) were effective against broadleaf weeds, while the narrow leaf weeds required a higher concentration of acetic acid (30 %).

Table 13: Comparison between efficacy of clove extract 10 % SL and acetic acid 36 % against *Phalares minor* under greenhouse conditions.

Materials	Concentrations %	% Efficacy of burning	% Reduction of shoots	
			Fresh weight	Dry weight
Clove extract 10 SL	4	95	65.2	50.5
	2	75	44.63	36.95
	1	63	16.4	26.63
Acetic acid 36 %	4	65	17.32	25.54

Data in table (14) and figure 4 showed the efficacy of clove extract 10 % SL and acetic acid against *Rumix dentatus* as pattern of dicotyledonous weeds under greenhouse condition, where clove extract SL formulation at 4 % gave the best efficacy appeared as burning and reduction in shoots fresh and dry weight, the efficacy values were 80, 58.53, and 61.53% respectively. However acetic acid at 4 % showed low efficacy against the tested weed, the percentage of efficiency burning and reduction in shoots fresh and dry weight were 60, 44.31 and 40.89 % after 14 days of application.

Table 14: Comparison between efficacy of clove extract 10 % SL and acetic acid 36 % against *Rumex dentatus* under greenhouse conditions

Materials	Concentrations %	% Efficacy burning	% Reduction of shoots	
			Fresh weight	Dry weight
Clove extract 10 SL	4	80	58.53	61.53
	2	70	52.69	57.08
	1	58	44.01	53.44
Acetic acid 36%	4	60	44.31	40.89

4. Conclusion

Polar components were extracted from clove with acetic acid, the chemical groups contained were determined phytochemically. Comparison between the effect of crude with solvent of extraction and the solvent alone was carried out biologically on testing on mono and dicotyledonous plants under laboratory conditions. Crude containing solvent showed higher efficacy on germination, root and shoot growth than solvent alone. The crude was formulated as 10 % (SL) and tested on the previously mentioned plants. It showed inhibition of germination, root and shoot growth of both plants by higher values compared to that of the crude with solvent. The new formula was also tested under greenhouse conditions on two different kinds of weeds, in both cases, it succeeded to burn and reduces shoot fresh and dry weight of both weeds but the effect on monocotyledons was higher than on dicotyledons. The new formulation could be used in weed control after completing the other required studies.

References

[1] Abbot's W. S. (1925). A method of Computing the Effectiveness of an Insecticide; *J. Econ. Ent.*, **18**: 265-267.

[2] Abdin O. A., Zhou X. M., Cloutier D., Coulman D. C., Faris M. A. & Smith D. L. (2000). Cover Crop and Interrow Tillage for Weed Control in Short Season Maize (Zea mays). *Eur. J. Agron*. **12**: 93-102.

[3] Abouziena H. F. & Haggag W. M. (2016). Weed Control in Clean Agriculture: A review, *Planta Daninha*, Viçosa-MG, **34(2)**: 377-392.

[4] Abouziena H. F. H., Omar A. A. M., Sharma S. D. & Singh M. (2009). Efficacy Comparison of Some New Natural-product of The Herbicide for Weed Control at Two Growth Stages. *Weed Technology*, **23**: 431-437.

[5] American Society of Testing Materials .ASTM. 2005. Standard Test Method for Rheological Properties of Non – Newtonian Materials by Rotational (Brookfield type) Viscometer, D-2196.

[6] American Society of Testing Materials. ASTM. 2001. Standard Test Method for Surface and Interfacial Tension Solution D-1331.

[7] Baker B. P., Grant J. A. & Malakar-Kuenen R. (2018). Cloves & Clove Oil Profile

[8] Active Ingredient Eligible for Minimum Risk Pesticide Use. New York State Integrated Pest Management Program. Cornell Cooperative Extension, http://hdl.handle.net/1813/56120, 1-15.

[9] Barbosa, A. D. (2014). An overview on the biological and pharmacological activities of saponins. Int. J Pharm Pharm Sci, vol 6, Isse 8, 47-50 Review article.

[10] Choi H., Sowndhararajan K., Cho N., Hwang K., Koo S., & Kim S. (2015). Evaluation of Herbicidal Potential of Essential Oils and their Components under in Vitro and Greenhouse Experiments. *Weed Turf. Sci.* **4(4)**:321-329.

[11] Cremlyn R. J. (1991). Agrochemicals . John Wiley & Sons, Chichester, UK.

[12] Dobrat W. & A. Martijn (1995). CIPAC Hand Book, vol. F, Collaborative International Pesticides Analytical Council Limited.

[13] El-Kady A. M. A., Betana D. M. & Hussein A. M. (2000). Evaluation of the Herbicidal Activity of the Active Extracts of *Eucalyptus Globutus* against Monocotyledon; *Egypt. J. Appl. Sci.*, **15(7)**: 565-576.

[14] Feroz, M., Ahmad, R., Sindhu, S. T. A. K. and Shahbaz, A. M. (1993). Antifangal activities of saponin from indigenous plant roots. Pak. Vet. J., 13: 44

[15] Finney D. J. 1952. Probit Analysis Statistical, 2nd Ed, Cambridge University.

[16] Fishel F. M. (2010). Pesticide Formulations. UF/IFAS EDIS Publication PI231. http://edis.ifas.ufl.edu/.

[17] Glauert,A. M., Dingle, J. A. and Lucy, J. A. (1962). Action of saponin on biological cell membranes. J Nature, 196: 953- 955.

[18] Handa S. S., Khanuja S. P. S., Longo G. & Rakesh D. D. (2008) Extraction Technologies for Medicinal and Aromatic Plants, (1stedn), no. 66. Italy: United Nations Industrial Development Organization and the International Centre for Science and High Technology.

[19] Herbicides. *Weed Sci.* **49**: 290-297.

[20] Hussein M. A. (1989). Relation between Physico-chemical Properties of Some Herbicide Combinations and Their Efficiency; M. Sc. Plant Protection Department, Faculty of Agriculture, AL-Azhar University.

[21] Kraehmer H., Laber B., Rosinger C., & Schulz A.(2014). Herbicides as Weed Control Agents: State of the Art: I. Weed Control Research and Safener Technology: The Path to Modern Agriculture, *Plant Physiology*. **166**: 1119-1131.

[22] Lynch M. I. & Griffin W. C. 1974. Food Emulsions in: Emulsion Technology, by Lissant K. J., Marcell Decker, Inc., New York. Mukerjee P. and K. J. Mysels (1971) Critical Micelle Concentration of Aqueous Surfactant Systems. National Bureau of Standards Washington DC, PP. 1-21.

[23] Nelson F. C. & Fiero G. W. 1954. A selected Aromatic Fraction Naturally Occurring in Petroleum as Pesticides Solvents; *J. Agric. Food Chem.*, **14(2)**: 1765-1737.

[24] Osipow L. I. 1964. Surface Chemistry Theory and Application. Reinhold Publishing Crop, New York, pp. 4736-4739.

[25] Powell G. R. & Spencer F. G. (1988). Photochemical Inhibitors of Velvet Leaf Germination as Models for New Biorational; Active Natural Products; ACS (Symp. American Chem. Soc.); Washington, D-C; 211-213.

[26] RAO S. (2000). Principles of Weed Science. 2^ed. NewYork:Science Publishers, 526 p.

[27] Ryckaert B., Spanoghe P., Haesaert G., Heremans B., Isebaert S. & Steurbaut W. (2007). Quntative Determination of the Influence of Adjuvant on Foliar Fungicide Residues; *Crop Protection*, **26**: 1589-1594.

[28] Santhi, K. and Sengottuvel, R.(2016). Qualitative and quantitative phytochemical analysis of *Moringa concanesis* Nimmo. Int. J. Curr. Microbiol. App. Sci. 5(1):633-640.

[29] Soltys, D., Rudzinska- Langwald, A.,Kurek, W., Gniazdowska, A., Sliwinska, E., and Bagatek, R.,(2011). Cyanamide mode of action during inhibition of onion (*Allium cepa* L.) root growth involves disturbances in cell division and cytoskeleton formation. Planta 234, 609- 621.

[30] Spanoghe P. D., Schampheleire, M., Van Dermeeren P & Steurbaut W. (2007) Influence of Agricultural Adjuvants on Droplet Spectra; *Pest Management Science*. **63(1)**: 416.

[31] Tawfik, H. Mona & El-Sisi, A. G. (1987). The Effect of Mixing Some Foliar Fertilizers on the Physical Properties and Insecticidal Activity of Some Locally Spray Oils against the Scale Insect *Parlatoria Ziziphus (Lucas).* 2^nd Nat. Conf. of Pests and Disease of Vegetable and Orchard, Ismailia, Egypt, pp: 367-376.

[32] Troyer J. R. (2001). In the beginning: The Multiple Discovery of The First Hormone.

[33] World Health Organization, WHO 1979. Specification of Pesticides Used in Public Health, 5^th ed. Geneva.

[34] Zabkiewicz J. A. (2000). Adjuvants and Herbicidal Efficacy - Present Status and Future Prospects. *Weed Research*, **40**: 139149, DOI: 10.1046/j.1365-3180.2000.00172.x.