Efficiency of Sodium Carbonate 90 % Soluble Powder Formulation on the Two-spotted Spider Mite *Tetranychus urticae* Koch under Laboratory Conditions.

Farag E. M[1*] and B. S. WAHBA[2]

1.Formulation Research Department, Central Agricultural Pesticides Lab. (CAPL), Agriculture Research Center (ARC), Dokki, Giza, Egypt

2.Plant Protection Research Institute, Agriculture Research Center (ARC), Dokki, Giza, Egypt.

*Correspondences at: Email: smfarag@yahoo.com

ABSTRACT: Sodium carbonate salt is formulated as a 90% soluble powder. All tests reported for this type of pesticide formulations were successful. The physicochemical properties of the sprayed solution were evaluated and the results showed improved physical and chemical properties that were highly correlated with the biological effectiveness of the new formulation. The newly formed soluble powder was tested under laboratory conditions on adults and hatching eggs of two-spotted spiders *Tetranychus urticae* Koch (Arachnida: Tetranychidae). As shown by the cumulative results of egg and adult hatching, this had a significant impact on egg and adult hatching. The EC_{50} values for hatching eggs and adults were 0.79 and 3259 (mg/ml), respectively. The new formula also showed high slope values for adults and hatching eggs.

KEYWORDS: Salts, Soluble Powders, Formulation and *Tetranychus urticae*

1.INTRODUCTION:

As one of the most polyphagous herbivores, the two-spotted spider mite (TSSM), *Tetranychus urticae* Koch (Acari: Tetranychidae), consumes around 1,100 plant species **(Bensoussan *et al.*, 2018)**, including over 150 plants of commercial importance **(Zhang, 2003)**. Gray or yellowing of the leaves is caused by feeding mites. Necrotic patches develop when leaves sustain injury. The opened flower is damaged by mites, causing the petals to brown and wilt, resembling spray burn. The two-spotted spider consumes sap, causing the mesophyll tissue to collapse and a little chlorotic spot to emerge at each feeding location. It is thought that between 18 and 22 cells are lost every minute. After a period of continuous feeding, the leaves wilt, discolor, and eventually turn yellow, gray, or brown **(Thomas and Denmark, 2016)**. According to **Stavrinides and Hadjistilla (2009)**, a large population of red spiders has the ability to completely destroy entire plants.

Conventional pesticides currently represent an indispensable means of combating some pests quickly, inexpensively and effectively **(Erdogan *et al.*, 2012)**. The continuous and repeated use of synthetic acaricide as a control agent to (TSSM) has caused a rapid emergence of chemical resistance to those that are sold commercially. An integrated, cost-effective mite control strategy has faced major challenges due to widespread mite resistance **(Cho *et al.*, 1995)**. Due to resistance to several known acaricides, it has become necessary to create and market substances with novel modes of action **(Devine *et al.*, 2001)**.

The active ingredient in numerous algaecide and fungicide treatments, sodium carbonate peroxyhydrate, was introduced as one of the main ingredients of sodium carbonate formulas produced as pellets. This active ingredient's final product functions as an oxidizing agent and destroys dangerous fungus and algae in particular. It is used in commercial greenhouses, garden centers, nurseries, and their warehouses, as well as outdoors to treat decorative plants, lawns, and landscapes (CAS No. 15630-89-4) **(Environmental Protection Agency, 128860)**. fact sheet). Another formulation of sodium carbonate was tested on *Aspergillus niger* and *Penicillium* to prevent mold on hard, nonporous surfaces **(Environmental Protection Agency (EPA), Reg. No. 72372-1, 2006)**.

The majority of pesticide formulations include a variety of inert components together with one or more active compounds. These inert elements are those that, in most cases, guarantee that the active agent reaches the parasite or target site and preserve the formulation's integrity **(Leifer, 1997)**.

A new step in our integrated pest management is to find safe and environmentally friendly active ingredients and formulate them into local formulations that can be used against the two-spotted spider after further recommended field testing. The aim of this work was to examine the effects of sodium carbonate, an inorganic salt, in a soluble powder formulation on the *Tetranychus urticae* Koch two-spotted spider in a lab setting.

2. MATERIALS AND METHODS:

2.1. Tested chemicals:

1) Sodium carbonate (molar mass 105.9888 $g{\cdot}mol^{-1}$), was supplied by EL-Gomhoria Co., Cairo, Egypt.

2) Surfactants: The supplier of surface-active agents was EL-Gomhoria Co. located in Cairo, Egypt.

2.2. physical and chemical properties of all formulation parts:

2.2.1. Active ingredient:

a) Solubility: The volume of distilled water, acetone, and xylene was measured in order to fully dissolve or mix one gram of the active component at 20 °C **(Nelson and Fiero, 1954)**. The following equation was used to calculate the % solubility:

W/V x 100

[Where V is the solvent volume for perfect solubility, and W is the weight of the active component].
b) Free acidity or alkalinity: **The WHO standards (1979)** method was followed during its implementation.

2.2.2. Surfactants:
a) Surface tension: In accordance with **ASTM D-1331 (2001)**, it was measured using a Du-Nouy tensiometer for solutions containing 0.5% (w/v) of a surfactant.
b) Free acidity or alkalinity: It was obtained as mentioned before.
c) Hydrophilic-lipophilic balance (HLB): A surfactant's hydrophilic-lipophilic balance (HLB) can be roughly determined by looking at how soluble it is in water **(Lynch and Griffin, 1974)**.
d) Critical micelle concentration **(CMC): Utilizing Osipow's (1964)** methodology, the concentration (CMC) at which the solution's surface tension remains constant upon additional surfactant concentration increase was ascertained.
e) Solubility: It was ascertained as previously stated.

2.2.3. Local formulated soluble powder:
a) Solubility: It was ascertained as previously stated.
b) Free acidity or alkalinity: It was measured using the previously mentioned methodology.
c) Accelerated storage: The chemical and physical stability of the new formulations was tested according to CIPAC M46.1 1995for three days at a temperature of 45 ± 2 °C.
2.2.4. Spray solution at recommended field dilution rate:
a) Electrical Conductivity: According to **(Dobrat and Martijn, 1995)**, it was computed using a Cole-Parmer pH/conductivity meter using the Cole-Parmer 1484-44, where µmhos is the unit of electrical conductivity.
b) Surface tension: As previously stated, a determination was made.
c) PH: It was determined by using the Cole-Parmer PH conductivity meter 1484-44 as per **(Dobrat and Martijn, 1995)**.
d) Viscosity: In accordance with **ASTM D-2196 (2005)**, the unit of measurement is centipoise, and it was computed using a Brookfield viscometer model DVII+Pro.

2.3. Bioassay:

2.3.1 Rearing of two-spotted spider mite:
The *T. urticea* mites used in this study came from our laboratory, where they were reared for more than 40 generations on *Acalypha marginata* at a temperature of 25 ± 2 °C, a relative humidity of 60 ± 10% and a light/dark cycle of 14:10 hours. At regular intervals, new fresh plants were added to the culture to keep it viable for research.

2.3.2 Under laboratory conditions:
2.3.2.1 Ovicidal efficacy:
using **(Hala, 2012)**, with minor modifications, the following caricidal effect of 90% sodium carbonate salt soluble powder on eggs of *T. Urticae* was tested: To test on egg stages, *Acalypha marginata* leaf discs with a diameter of 2.5 cm were submerged in five concentrations (10, 100, 1000, 10000, and 100000 ppm) for ten seconds. The residual solution was then dried using filter paper. Hairbrushes on the underside of leaf discs were used to transfer day-old eggs. Three times (50 eggs each) of each concentration and control were reproduced. The control was made using water. Eggs were maintained in a lab environment, and the hatchability of each concentration was tracked for 72 hours after treatment.

2.3.2.2 Efficacy on individuals:
The leaf disc test was conducted using the methodology outlined in the Pree *et al*., 1989 procedure. *Acalypha marginata* leaf discs of 2.5 cm in diameter were dipped into progressively higher concentrations of the preparation (10, 100, 1000, 10,000, and 100,000 ppm). The deadly concentration of the active ingredient is given in parts per million, based on trials conducted with six concentrations of each formulation. The slices were kept at a consistent temperature and humidity of 25 ± 2 °C, 65 ± 5% relative humidity, and a 16:8 photoperiod in a Petri dish with moist cotton on top. Ten adult female *T. urticae* (1 day old) were used, with a tiny brush, on every slice. Distilled water was utilized to submerge the control slices. Tests were conducted in triplicate for every preparation's concentration 48 hours following treatment, the mortality of mites treated with the formulation was evaluated.

2.4. Statistical analysis:

Using the **Abbott equation (1925)**, the inhibition percentages were adjusted and the concentration inhibition regression line was obtained with **Finney (1952)**.

3. RESULTS AND DISCUSSION:

3.1. Preparation of sodium carbonate as soluble powder formulations (SP):

Although soluble powders are rare, their rarity is due to the fact that few solid active ingredients are soluble in water; Those that are soluble powders (and therefore formulated as soluble powders) are then mixed with water in the spray container where they dissolve and form a true solution before being sprayed. According to **Anderw *et al.*, (2011)**, soluble powders do not cause abrasion on application machines.

3.2. physical and chemical properties of sodium carbonate as active ingredient:

The physical and chemical characteristics of the active ingredient sodium carbonate are listed in Table 1. Although it showed only slight solubility in water, it showed completely insolublity in two organic solvents, acetone and xylene. It also exhibited an alkaline quality, as evidenced by the measured pH and alkalinity values. The data showed that sodium carbonate can be prepared as a soluble powder.

3.2.1. Physico-chemical properties of surface-active agents:
Three surfactants symbolled (A, B and C) were tested for their possible use as surfactants in the preparation of this salt in the form of a soluble powder (Table: 2). All were insoluble in xylene, but A had the greatest solubility in

Table (1): Physical and chemical properties of sodium carbonate as active ingredient

Physical property Active ingredient	Solubility % (W/V)			Free alkalinity as % NaOH	PH
	Water	Acetone	Xylene		
Sodium carbonate	10	N.S*	NS	52.013	11.37

N.S*: means insoluble.

acetone and water (40 and 56), followed by B, which had the same solubility in both solvents, and C, which had a solubility of 25 and 33.3%, respectively. In addition, they all had a lower surface tension than water (72 dyn/cm). When their HLB-values were tested, all showed values above 13, suggesting that they could all be used as dispersants. In addition, they showed identical free acidity (0.05, acids with week property) and relative acidic pH values. However, their CMC values were significantly different, with A, B, and C having values of 0.2%, 0.4%, and 5% respectively.

Table (2): Physical and chemical properties of the tested surfactants

Surface active agent	Solubility % (W/V)			Surface tension dyne/cm	HLB	CMC %	Free acidity as % H_2SO_4	PH
	Water	Acetone	Xylene					
A	40	56	N.S*	43	> 13	0.2	0.05	3.99
B	33.3	33.3	N.S*	53	> 13	0.4	0.05	3.79
C	25.1	33.3	N.S*	54	> 13	0.5	0.05	6.08

3.3. Physical and chemical properties of new locally prepared 90 % soluble powder formulation after different storage circumstances:

The new sodium carbonate 90% soluble powder was alkaline before storage, which was proven by measuring its free alkalinity, which was found to be 33.6. Furthermore, it showed a surface tension value of 47.2 dynes/cm instead of 72 dynes/cm of water. Solubility and sedimentation tests showed that the solubility in water is excellent, no sedimentation was detected. It had a sodium hydroxide-free alkalinity of 34.3, a surface tension of 47.2 dyn/cm, excellent water solubility and no precipitation during accelerated storage. It can be concluded that sodium carbonate newly prepared formulation in the form of 90% soluble powder is able to maintain its physicochemical properties before and after accelerated storage **(El-Sharkawy *et al.*, 2020)**.

Table (3): Physical and chemical properties of new locally prepared 90 % soluble powder formulation after different storage circumstances

Formulation	Before storage				After storage			
	Free alkalinity as % NaOH	Surface tension dyne/cm	Solubility	Sedimentation	Free alkalinity as % NaOH	Surface tension dyne/cm	Solubility	Sedimentation
Sodium carbonate	33.6	47.2	soluble	Nil	34.4	47.2	soluble	Nil

3.4. Physical and chemical properties of spray solution:

The most important factor for the biological effectiveness of the prepared formulation is the physicochemical features of the spray solution. The newly obtained soluble powder formulation showed a low surface tension with respect to water (47 and 72 dyn/cm, respectively), indicating high activity according to Osipow, 1964, who noted that the diffusion and wettability of a surface treated with a pesticide increased this is because the pesticide solution's surface tension is lowered, increasing the pesticide's efficacy. It was claimed that increasing the electrical conductivity of the spray solution would cause insecticides to deionize, increase their deposition and penetration into the tested surface, and ultimately improve the effectiveness of the insecticide **(Twifik and El-Sisi, 1987)**. This high electrical conductivity (638 µmhos) could lead to better effectiveness of the pesticide. The new formula was characterized by high viscosity values (9.98 and 9.97 centipoise), which can increase retention adhesion and cause reductive drift, increased viscosity has also been linked to increased pesticide activity **(Richardson, 1974)**. Table (5) displayed the effect of 90% soluble powder sodium carbonate formulation on adults of the two-spotted spiders under laboratory conditions at a range of concentrations. After 48 hours of treatment, accumulation toxicity was detected at 100, 1,000, 10,000 and 100,000 ppm. 20, 35, 51 and 93 .1%, respectively, indicating a gradual increase in toxicity with increasing concentration.

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

Property / Formulation	Viscosity centipoise	Electrical conductivity µ mhos	PH	Surface tension dyne/cm
Sodium carbonate	9.98	638	13.5	47

Table (5): The effect of sodium carbonate 90 % soluble powder on adults of the two-spotted spider mites adults under laboratory conditions.

Concentration (ppm) / Day	10	100	1000	10000	100000
1	0.0	20	11	0.0	93.1
2	0.0	0.0	24	51.3	0.0
A.T*	0.0	20	35	51.9	93.1

A.T*: The overall amount of toxicity for each concentration at the conclusion of the experiment.

The effect of 90% soluble powder sodium carbonate formulation at various concentrations on the spider mite hatched eggs was shown in Table (6). According to the obtained results, the accumulation hatchability values were 93, 76.7, 50, 20 and 10% at concentrations of 10, 100, 1000 and 10000 ppm, respectively. Additionally, there were values of 3.8, 26.7, 48.3, and 73.3 and an 89.7% inhibition percentage at the same concentrations.

Table (6): The effect of sodium carbonate 90 % soluble powder on egg hatching of TSSM under laboratory conditions

Concentration (ppm) / Day	0.0	10	100	1000	10000	100000
1	23	23	50	23	3.3	0.0
2	33.7	33	30	20.3	9.7	0.0
3	40	37	0.0	6.7	7	10
A.H*	96.7	93	76.7	50	20	10
% I	0.0	3.8	26.7	48.3	73.3	89.7

A.H*: Accumulation hatchability: the total amount of hatching for each concentration at the conclusion of the experiment.
% I: The percentage of inhibition.

The soluble powder formulation of sodium carbonate was tested under laboratory conditions on adult and hatched eggs of the TSSM and the results were previously presented. A LDP line parameters of the new soluble powder formulation was considered. Since the EC_{50} for eggs hatching and adult was 797.9 and 3,259 ppm, respectively, the sodium carbonate newly prepared formulation was effective for both hatched eggs and adults. The slope value also showed higher effectiveness of the new sodium carbonate soluble powder in hatching eggs and lower effectiveness in adults.

Table (7): EC_{50} and slope for sodium carbonate soluble powder on adults and hatched egg of the TSSM under laboratory conditions

Local formulation	EC_{50} ppm (mg/ml)		Slope	
	Egg	Adults	Egg	Adults
Sodium carbonate (SP)	0.79	3.2	1.02	0.76

CONCLUSION:

The sodium carbonate salt was formulated as a 90% soluble powder that met the physicochemical properties of soluble powders and was tested for TSSM under lab. conditions. It showed remarkable values for inhibition of hatching of eggs and adults as their EC_{50} values obtained were 0.79 and 3.2 mg/ml, respectively.

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دراسة كفاءة المسحوق القابل للذوبان فى الماء بتركيز 90 % لملح كربونات الصوديوم على الأكاروس تحت ظروف المعمل.

السيد محي الدين فرج[1] - باسم صبري وهبه[2]

1.قسم بحوث مستحضرات المبيدات – المعمل المركزي للمبيدات - مركز البحوث الزراعية - دقى - جيزة - مصر

2.معهد بحوث وقاية النباتات - مركز البحوث الزراعية - دقى - جيزة - مصر.

الملخص العربي:

تم تجهيز ملح كربونات الصوديوم فى صورة مسحوق قابل للذوبان فى الماء بتركيز 90 % وتم اجراء الاختبارات الطبيعية والكيميائية المحددة لمستحضرات المساحيق القابلة للذوبان فى الماء فاجتازها المستحضر الجديد بنجاح. كما درست الخواص الطبيعية والكيميائية لمحلول الرش والتي أظهرت تحسنا ملحوظا وهو ما يرتبط ارتباطا وثيقا بالكفاءة البيولوجية للمستحضر الجديد . ثم تم تجريب المستحضر الجديد على فقس البيض والأفراد البالغة للأكاروس تحت ظروف المعمل. أظهرالمستحضر تأثيرا ملحوظا على تثبيط فقس البيض وكذلك على الأفراد وهو ما اتضح من قيم التركيز النصفى المميت حيث كانت 9.7 ، 3.2 ملجم/ملم لفقس البيض والأفراد على الترتيب. بالإضافة إلى ذلك، أظهرالمستحضر الجديد قيم ميل عالية لكل من فقس البيض والأفراد، حيث كانت تلك القيم 1.02 و 0.76 على الترتيب.