ENCAPSULATION OF NPK MINERAL FERTILIZER GRANULES BY DIFFERENT TYPES OF ALKYD RESINS

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Abstract

Three different kinds of alkyd resins were used for encapsulation a commercial granules fertilizers NPK (N 12%, P_2O_5 8% and K_2O 18%) with particle diameter ranges from 3 to 5 mm. The total concentration of the alkyd coated film was less than 10 % of the total weight of the fertilizer composition. The release rate was measured by conductivity test. The area of the coating film was determined by mathematical calculations. Controlled release parameters including membrane thickness, granule radius and type of resin were taken into consideration.

Keywords: Controlled-release fertilizer, Alkyd resins, Encapsulation NPK

1. Introduction

Controlled-release fertilizers (CRF) are made to release their nutrient contents gradually and to coincide with the nutrient requirement of the plant [1]. These fertilizers can be physically prepared from the granules of the soluble fertilizers by coating them with the materials which reduce their dissolution rate. The use of controlled-release fertilizers causes an increase of their efficiency reduce soil toxicity [2]. The coating materials used must be cheap and biodegradable. Most of the previous research works are based on encapsulation of fertilizers by polysulfone [3-5], polyolefin's [6-8], polystyrene, polyurethane [9,10], polyacryloamide [11], derivatives of cellulose and many others. In our study we used environmentally friendly three types of alkyd resin. The compositions of alkyd resins [12] are based on vegetable oil-glycerol and dibasic acid; the advantage of alkyd resins, is its biodegradability especially long oil length alkyd. The release rate from coated granules was determined by conductivity test [13].

2. Experimental

2.1. Materials

Commercial soluble granules NPK fertilizer was obtained from producer Yara porsgrum company (Norway), Implorer Yara Agrotrade A.R.E. Three types of alkyd resins from Egyptian American Paint and Coating Company: Long soya alkyd resin; code (S421-N 6), short mod. soya alkyd resin; code (S 136-N 6) and styrenated alkyd resin; code (B 7445 M).

2.2. Instruments and Apparatus

a. Infrared spectra were recorded on a Nicolet 20DX-FTIR spectrometer with a resolution of 2 cm^{-1} and a minimum of 20 cumulative scans.

b. Conductivity/TDS meter Model YK-22CT Lutron Electronic made in Canada.

c. Atomic Absorption Model Perkin Elimer A Analyst 200.

2.3. Methods

Preparation of standard curve; (0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9 and 1.0 g) of salt of commercial fertilizers was dissolved after grinding in 50 ml distilled water and allow to stand for about 24 hrs.; then the relation between Electrical Conductivity (EC) and Total Dissolved Salt (TDS) was determined.

Encapsulation of the commercial fertilizers granules

The different kinds of alkyd resins were diluted to 20% and 40% concentration by the suitable solvent. About 20 g of the commercial granules of fertilizers were soaken in diluted resin for different times of 10, 20 or 30 minutes. Then the granules were separated by filtration and allowed to dry at room temperature for about 6 hrs. to get rid of solvent then heated in oven at 80 - 90 C° until constant weight. The difference between the weight of granules before and after coating of the alkyd resin was determined.

The experimental conditions for experiments no. 1-29; for the different types of alkyd are tabulated in table (1).

Table 1: Experimental conditions of different types of alkyd resins.

.No. of Exp	Type of alkyd	Size of granules	Dipping time (min.)	Conc. of resin (wt/wt)
and 3 2 ,1	Styrenated	mm 5	and 30 20 ,10	% 20
4	Styrenated	mm 5	30	% 40
and 7 6 ,5	Styrenated	mm 4	and 30 20 ,10	% 20
and 10 9 ,8	Styrenated	mm 3	and 30 20 ,10	% 20
and 13 12 ,11	Short	mm 5	and 30 20 ,10	% 20
14	Short	mm 5	30	% 40
and 16 15	Short	mm 4	and 20 10	% 20
and 19 18 ,17	Short	mm 3	and 30 20 ,10	% 20
and 22 21 ,20	Long	mm 5	and 30 20 ,10	% 20
23	Long	mm 5	30	% 40
and 26 25 ,24	Long	mm 4	and 30 20 ,10	% 20
and 29 28 ,27	Long	mm 3	and 30 20 ,10	% 20

3. Results And Discussion

3.1. Analysis of commercial fertilizers: The granules were analyzed to know the percent of N, P_2O_5 , K_2O, and any other additives present other than the principle salt. N; 11.61 % (by using kjeldahl apparatus), P_2O_5; 8.74 %, K_2O; 17.88 %, MgO; 0.17 %, also analysis of trace element as manganese; 0.007%, zinc; 0.003%, calcium; 0.283% and copper; 0.002% (by using Atomic Absorption apparatus) , elemental sulfur is 7.99 % and kaolin about 11.91 %.

Table 2: Specification of alkyd resins

Type of resin	Solid content	Acid value
Long alkyd resin	70 %	7
Short alkyd resin	60 %	12
Styrenated alkyd resin	60 %	6

3.2. Interpretation of FTIR of the three types of alkyd resins:-

1. Long soya alkyd resin:

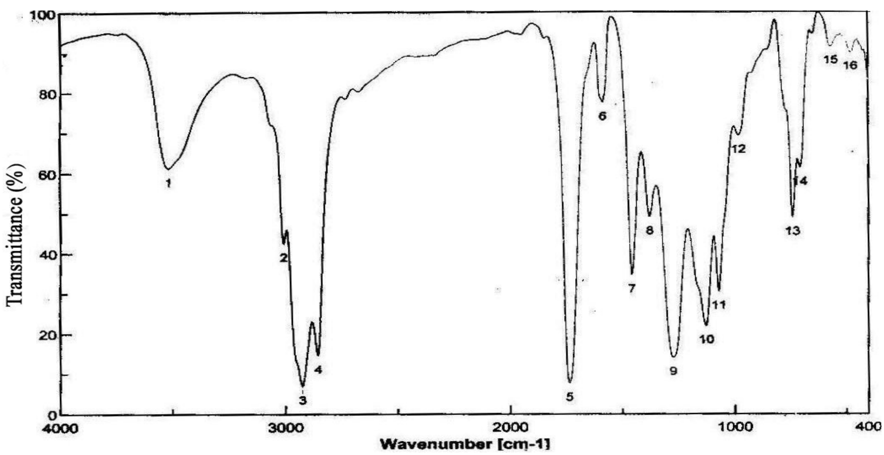

Fig.1: FTIR spectra of long oil soya alkyd resin

Table 3: The various peaks assigned for long soya alkyd resins

Peaks	Bands (cm^{-1})	Assignment
1	3518	O – H group.
2	3006	Aromatic C-H of xylene solvent.
3&4	2925 &2856	C-H Stretching vibration.
5	1738	C = O Stretching vibration.
6	1588	C=C (aliphatic and aromatic) stretching band.

2. Short mod. soya alkyd resin:

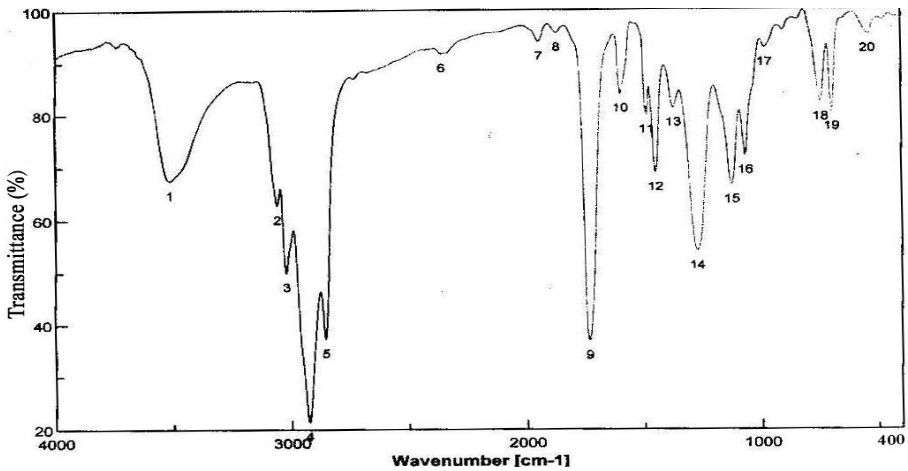

Fig. 2: FTIR spectra of short mod. soya alkyd resin

Table 4: The various peaks assigned for short mod. soya alkyd resin

Peaks	Bands (cm^{-1})	Assignment
1&2	3743 &3518	O – H group.
3&4	3066 &3005	Aromatic C-H of xylene solvent.
5&6	2925 &2856	C-H Stretching vibration.
7&8&9	2362 &2097 &1957	–C≡C– stretching vibration.
10	1728	C = O Stretching vibration.
11	1593	C=C (aliphatic and aromatic) stretching band.

3. Styrenated alkyd resin:

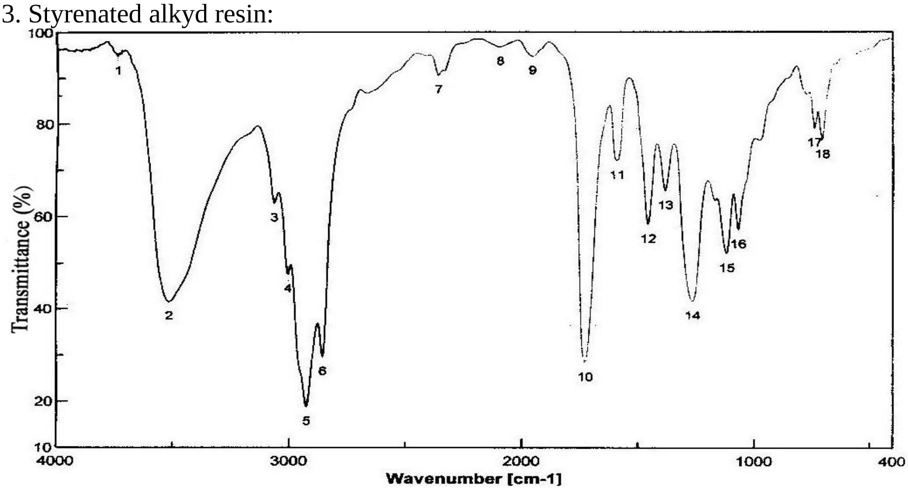

Fig. 3: FTIR spectra of styrenated alkyd resin

Table 5: The various peaks assigned for styrenated alkyd resin

Peaks	Bands (cm^{-1})	Assignment
1	3515	O – H group.
2&3	3060 &3022	Aromatic C-H of xylene solvent.
4&5	2925 &2855	-C-H Stretching vibration.
6&7	2360 &1947	–C≡C– stretching vibration.
8&9	1873 &1735	C = O Stretching vibration.
10	1600	C-C,C=C (aliphatic and aromatic) stretching band.

3.3. Determination of the film thickness of different alkyd resins covering the fertilizers granules with different diameters (3, 4 and 5 mm).

Weigh a glass plate and then using a film applicator to cover an area of 100 cm^2. After the drying of the varnish film, the weight of the dry varnish represents the weight needed to cover 100 cm^2 with a certain thickness. The film thickness was determined by thickness gauge.

The surface area of 100 g of balls of diameter 5 mm is equal to 1492.92 cm^2; in case of 4 mm is equal 1659.5 cm^2 and in case of 3 mm is equal 1980.09 cm^2.

Examples: Short mod. Soya Alkyd resin:- 0.2406 g covers 100 cm^2 with thickness 25 µm. The surface area of 100 g granules with diameter 3 mm equal 1980.09 cm^2. Therefore 1980.09 cm^2 will need 4.7640 g to give film thickness 25 µm. The surface area of 100 g granules with diameter 4 mm equal to 1659.504 cm^2. Since 100 cm^2 need 0.2406 g alkyd for thickness 25 µm. So 1659.504 cm^2 will need 3.9927 g with thickness 25 µm. For diameter 5 mm the surface area of 100 g ball equal 1492.92 cm^2. In this case of 3.592 g will cover the surface with thickness 25 µm.

In conclusion; (i) For short mod. soya alkyd resin the surface area of 100 g balls of diameter 3 mm will need 4.7640 g resin to give 25 µm thickness and 3.9919 g, of diameter 4 mm and 3.5919 g, of 5 mm for the same thickness. (ii) For styrenated alkyd resin 100 cm^2 need 0.4116 g resin for 25 µm film thickness according to the previous calculation. 100 g balls with diameter 3 mm will need 8.1496 g resins for 25 µm film thickness, for 4 mm diameter, the 100 g ball will need 6.83 g, for 5 mm diameter will need 6.14 g resin. (iii) For long alkyd resin 100 cm^2 need 0.2677 g resin to obtain 25 µm film thickness according to the previous calculation. 100 g balls with diameter 3 mm will need 5.3 g resins for 25 µm film thickness. For 4 mm diameter, the 100 g ball will need 4.42 g for 5 mm diameter will need 4 g resin.

3.4. Standard curve

Table 6: Effect of total dissolved salt for commercial fertilizers on EC.

Wt of Salt (g)	EC (µs/cm)	TDS (mg/l)	Factor A
0	0	0	0
0.1	2310	1533	0.663
0.2	4320	2867	0.663
0.3	6530	4340	0.664
0.4	8160	5407	0.662
0.5	10020	6607	0.669
0.6	11280	7520	0.666
0.7	12820	8527	0.665
0.8	14420	9613	0.666
0.9	15700	10467	0.666
1.0	16950	11300	0.666

Where Factor A= TDS/EC

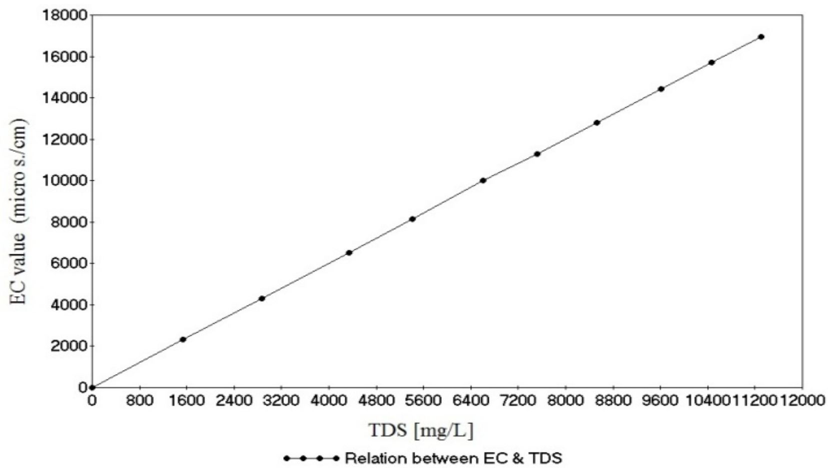

Fig. 4: Standard curve between EC & TDS

3.5. Encapsulation of the granules of the fertilizers using three different kinds of alkyd resins

Tables indicate the effect of granules particle size and resin film thickness on the release rate of fertilizers as function of time at room temperature for styrenated alkyd resin. Results are tabulated in tables (7 – 10) and illustrated in figs. (5 – 7).

Short alkyd resin for encapsulation the fertilizer granules. Results are tabulated in tables (11 – 13) and illustrated in figs. (8 – 10).

Long alkyd resin for encapsulation the fertilizer granules. Results are tabulated in tables (14 – 16) and illustrated in figs. (11 – 13).

Experiment (1)

Type of resin: Styrenated alkyd resin

Size of granules 5 mm, weight percent of resin 2.90 %, film thickness 11.8 μm.

Table 7: Relation between time, EC, TDS and Factor A.

Time (min.)	EC (μs/cm)	TDS (mg/l)	Factor A
0	0	0	0
5	4990	3327	0.666
10	7610	5073	0.666
15	9600	6400	0.666
20	11270	7507	0.666
25	12830	8553	0.666
30	14120	9413	0.666
35	14890	9920	0.666
40	15370	10240	0.666
45	15770	10507	0.666
50	16160	10787	0.667
55	16410	10956	0.667
60	16600	11007	0.663
65	16600	11007	0.663

Type of resin: Styrenated alkyd resin, size of granules: 5 mm.

Table 8: Relation between time, EC, TDS and Factor A.

No. of Exp.	Wt. percent of resin	Film Thickness (μm)	Time of Leaching (min.)	EC (μs/cm)	TDS (mg/l)	Factor A
1	2.90	11.8	60	16600	11007	0.663
2	2.54	10.5	50	16600	11053	0.665
3	2.38	10	45	16600	11007	0.663
4	7.18	29.2	320	15350	10233	0.666

From the previous data as a result of experiments 1 to 3, it can be noticed that the rate of leaching is decreased as the film thickness increases which lead to the decrease of the porosity of the alkyd resin film. In case of exp. 4 the film thickness is 29.2 μm and the total time of leaching equal to 320 minutes.

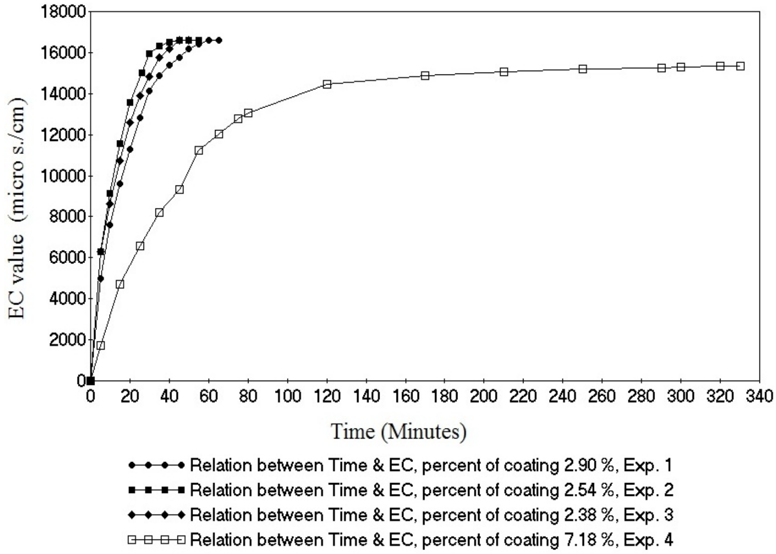

Fig. 5: Relation between Time & EC for exp. 1 to 4.

Type of resin: Styrenated alkyd resin, size of granules: 4 mm.

Table 9: Relation between time, EC, TDS and Factor A.

No. of Exp.	Wt. percent of resin	Film Thickness (µm)	Time of Leaching (min.)	EC (µs/cm)	TDS (mg/l)	Factor A
5	3.59	13.2	50	16060	10900	0.680
6	3.87	14.2	45	16100	10740	0.667
7	3.05	11.2	35	16400	10950	0.667

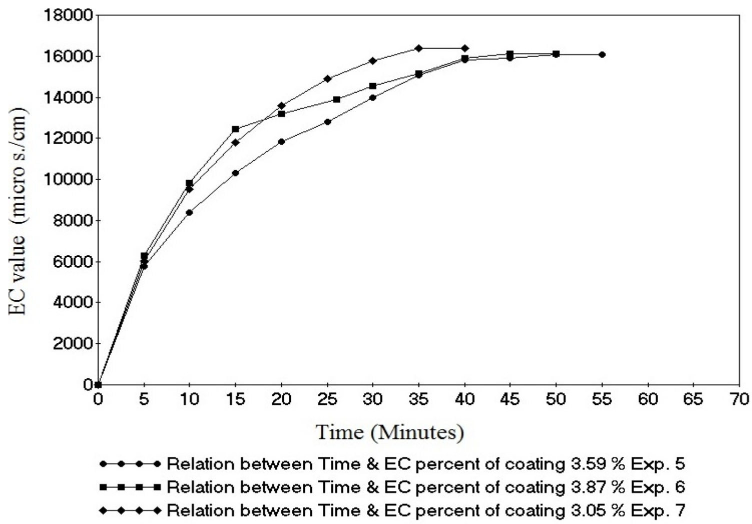

Fig. 6: Relation between Time, EC for exp. 5 to 7.

Type of resin: Styrenated alkyd resin, size of granules: 3 mm.

Table 10: Relation between time, EC, TDS and Factor A.

No. of Exp.	Wt. percent of resin	Film Thickness (μm)	Time of Leaching (min.)	EC (μs/cm)	TDS (mg/l)	Factor A
8	3.52	11.0	40	16100	10787	0.670
9	3.77	11.6	60	16300	10866	0.666
10	3.98	12.2	50	16270	10846	0.666

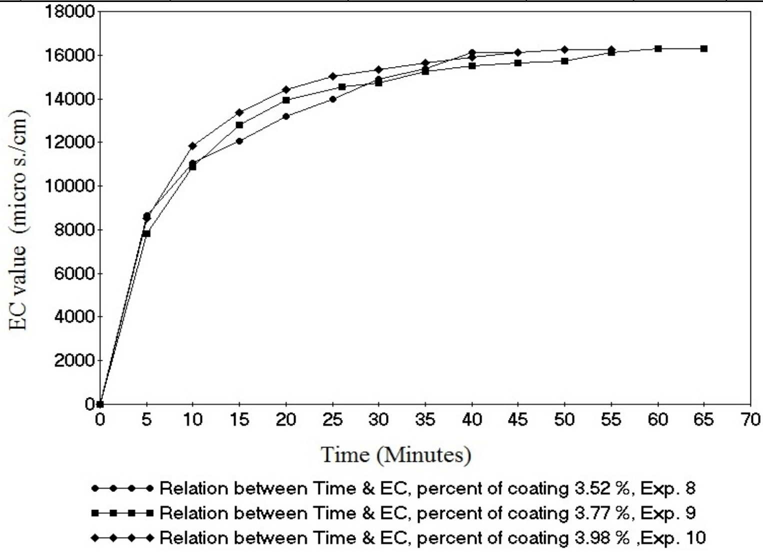

Fig. 7: Relation between Time, EC for exp. 8 to 10.

Experiments (5 – 10) clearly indicated that both porosity and granule radius have a significant effect on the leaching rate. Also the results given in tables (9 – 10) show that not only the film thickness (porosity) and granule radius affect the rate of release but also the type of alkyd resins. As a matter of fact in case of styrenated alkyd the leaching rate is faster than that in case of using short or long alkyd resin with the same film thickness and granule diameter. This is due to the tackiness present in both short and long alkyd which has a role to decrease the rate of solubility of the fertilizer.

Type of resin: Short alkyd resin, size of granules: 5 mm.

Table 11: Relation between time, EC, TDS and Factor A.

No. of Exp.	Wt. percent of resin	Film Thickness (μm)	Time of Leaching (min.)	EC (μs/cm)	TDS (mg/l)	Factor A
11	2.85	20	65	16400	10933	0.666

12	3.35	23.3	125	16300	10866	0.666
13	2.72	20	80	16400	10933	0.666
14	6.12	42.6	260	15980	10653	0.666

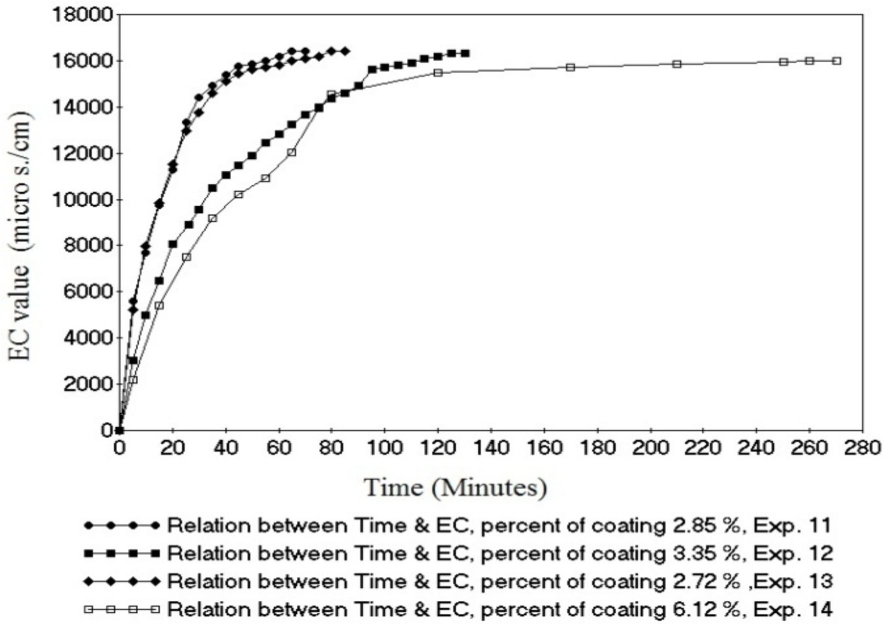

Fig. 8: Relation between Time & EC for exp. 11-14.

Type of resin: Short alkyd resin, size of granules: 4 mm.

Table 12: Relation between time, EC, TDS and Factor A.

No. of Exp.	Wt. percent of resin	Film Thickness (µm)	Time of Leaching (min.)	EC (µs/cm)	TDS (mg/l)	Factor A
15	3.15	20	45	16400	10933	0.666
16	3.72	23.3	55	16300	10866	0.666

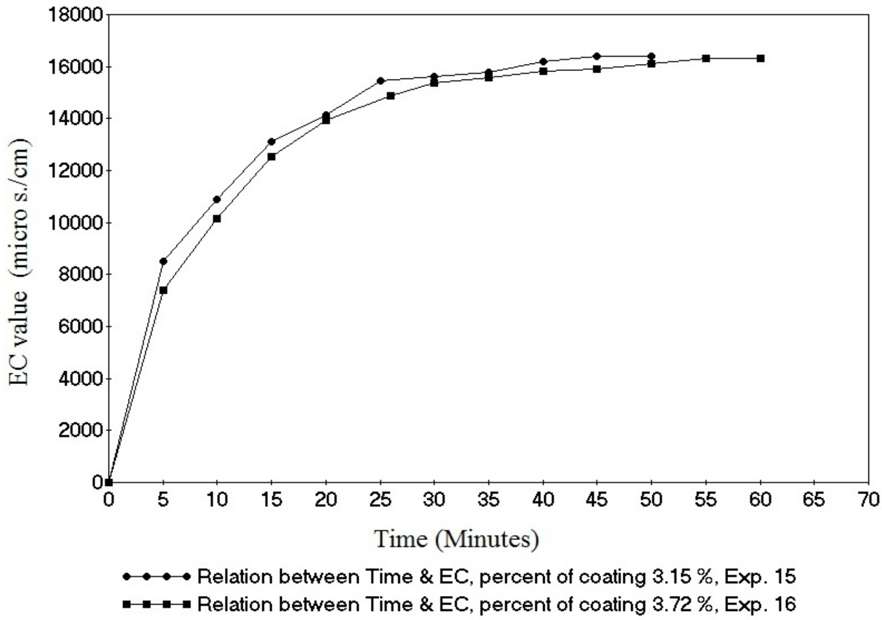

Fig. 9: Relation between Time, EC for exp. 15 and 16.

Type of resin: Short alkyd resin, size of granules: 3 mm.

Table 13: Relation between time, EC, TDS and Factor A.

No. of Exp.	Wt. percent of resin	Film Thickness (µm)	Time of Leaching (min.)	EC (µs/cm)	TDS (mg/l)	Factor A
17	3.86	20.25	60	16280	10853	0.666
18	3.72	19.5	50	16280	10853	0.666
19	3.14	16.5	45	16410	10939	0.666

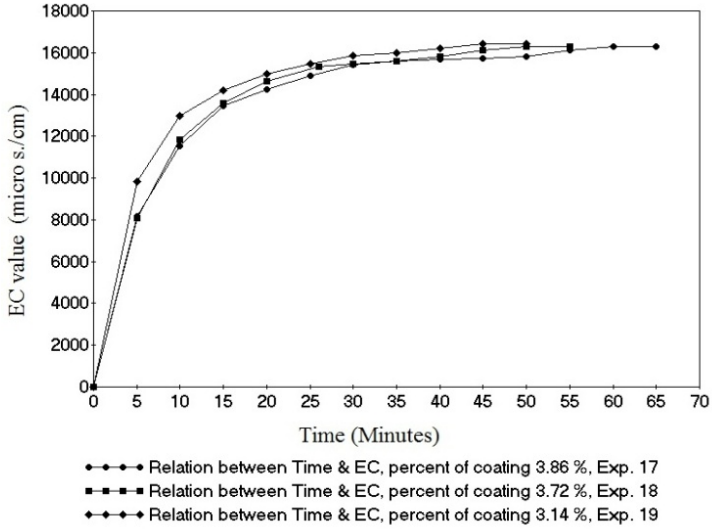

Fig. 10: Relation between Time, EC for exp. 17 to 19.

Type of resin: Long alkyd resin, size of granules: 5 mm.

Table 14: Relation between time, EC, TDS and Factor A.

No. of Exp.	Wt. percent of resin	Film Thickness (µm)	Time of Leaching (min.)	EC (µs/cm)	TDS (mg/l)	Factor A
20	2.73	13	45	16400	10933	0.666
21	2.96	14	45	16400	10933	0.666
22	2.47	15.5	60	16530	11019	0.666
23	5.40	34	190	16130	10753	0.666

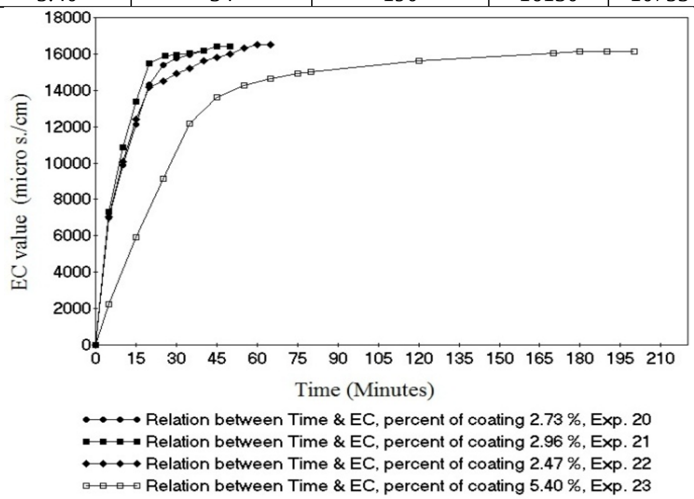

Fig. 11: Relation between Time & EC for exp. 20 to 23

Type of resin: Long alkyd resin, size of granules: 4 mm.

Table 15: Relation between time, EC, TDS and Factor A.

No. of Exp.	Wt. percent of resin	Film Thickness (μm)	Time of Leaching (min.)	EC (μs/cm)	TDS (mg/l)	Factor A
24	3.65	20.6	55	16330	10886	0.666
25	2.86	16	35	16460	10973	0.666
26	3.02	17	50	16510	11000	0.666

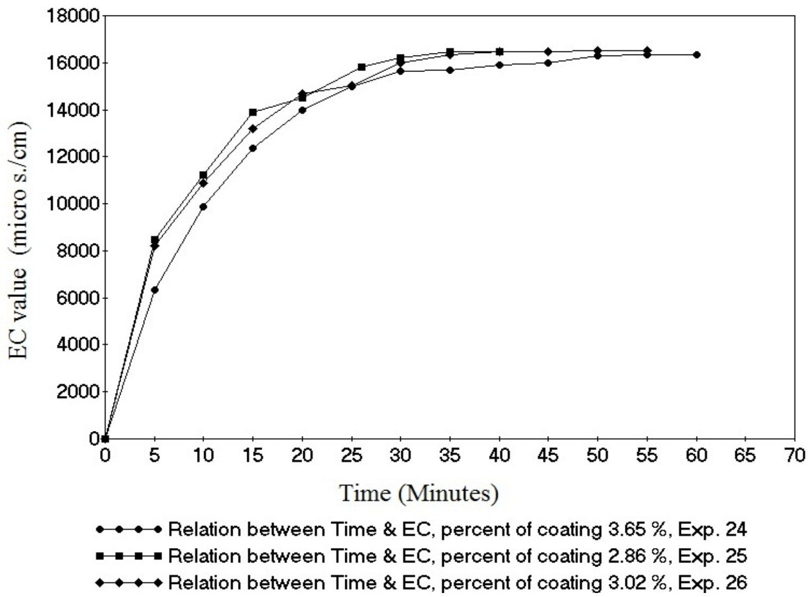

Fig. 12: Relation between Time, EC for exp. 24 to 26.

Type of resin: Long alkyd resin, size of granules: 3 mm.

Table 16: Relation between time, EC, TDS and Factor A.

No. of Exp.	Wt. percent of resin	Film Thickness (μm)	Time of Leaching (min.)	EC (μs/cm)	TDS (mg/l)	Factor A
27	3.85	18.2	65	16280	10853	0.666
28	3.74	17.7	40	16130	10760	0.667
29	3.90	18.5	35	16060	10707	0.666

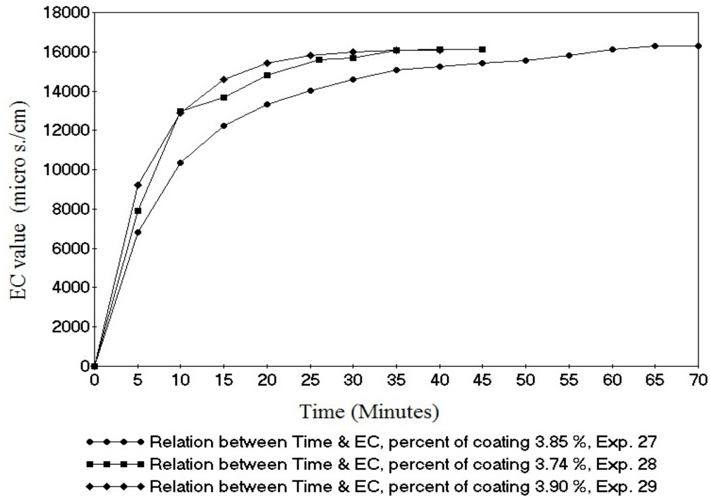

Fig. 13: Relation between Time, EC for exp. 27 to 29.

3. Conclusions

1. The method used for encapsulation of fertilizers in polymeric coatings proved to be effective in reducing fertilizer losses and to minimize environmental pollution. The release rate from the coated fertilizer granules was determined by conductivity test.
2. The area of the coating film was determined by mathematical calculation. The thickness of the coated film was determined by spreading a known weight of alkyd resin to cover the surface area of the granules by film applicator on a glass plate, then determine the weight and area of the dried film and measure the thickness by thickness gauge.
3. Controlled release parameters including membrane thickness, granule radius and type of resins were studied. It was found that the rate of leaching is decreased as the film thickness increases which lead to the decrease of the porosity of the alkyd resin film.
4. The most significant observation drawn from this investigation indicates that the best alkyd resin is long oil alkyd to be used for its low price and environmentally friendly due to its biodegradation.

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الملخص العربى

يهدف البحث إلى اعداد أسمدة شحيحة الذوبان والتي تعطى تغذية مستمرة للنبات بدلاً من استخدام أسمدة تقليدية سريعة الذوبان والتي ينجم عنها سمية النباتات من الزيادة الهائلة في تركيز ايونات العناصر في التربة وذلك بطلاء السماد بموادراتنجية للحد من خروج العناصر بسرعة هائلة والتحكم في خروجها في مدد طويلة يستفيد منها النبات وتقلل من كمية السماد المستخدم نظراً لحفاظه على العناصر أطول وقت ممكن.

تم استخدام طريقة تغليف السماد بطلاء راتنجى للتقليل من فقدان السماد وعدم الاستفادة منة وتعتبر هذه الطريقةعملية لترشيد الاستخدام وللتقليل من التلوث البيئي والتأثير على تربة النبات. وتم استخدام ثلاثة أنواع من راتنجات الألكيد لتغليف حبيبات سماد تجارى محبب يحتوى على (عنصر النيتروجين 8 % وعنصر الفسفورفي صورة خامس أكسيد الفسفور 12 % وعنصر البوتاسيوم في صورة أكسيد البوتاسيوم 18 % وأيضا يحتوى على عناصر صغرى وقابل للذوبان في الماء) و يتراوحقطر الحبيبات منة 3 مم إلى 5 مم. وتم مراعاة ألا تزيد نسبة راتنج الألكيد في الخلطة عن 10 % من وزن الخليط. ثم قياس نسبة ذوبان أملاح السماد في الماء باستخدام جهاز التوصيلية الكهربائية.

تم تعيين مساحة طبقة الطلاء بطريقة حسابية ثم تعيين سمك الطلاء وذلك بفرد وزنةمن الألكيد اللازمة لتغليف سطح الحبيبات على سطح زجاجي باستخدام جهاز فيلم ابليكاتوروقياس سمك الفيلم باستخدام جهاز خاص بعد جفاف الطلاء. وقد لوحظ إن عملية معدل إفراز السماد تعتمد على ثلاثة عوامل هي سمك طبقة الطلاء وقطر الحبيبات ونوع الألكيد. وقد لوحظ أنه في حالة الطلاء باستخدام راتنجات الألكيد المعدلة بالاستيرين تكون عملية الإفراز أسرع منه في حالة الطلاء باستخدام راتنج الألكيد القصير الزيت أو الألكيد الطويل الزيت بنفس سمك طبقة الطلاء ونفس قطر الحبيبة وذلك يرجع لأنه في حالة الألكيد الطويل الزيت أو القصير الزيت يكون التصاق الحبيبات شديد ويؤدى ذلك إلى زيادة وقت ذوبان السماد. و بزيادة قطر حبيبات السماد تزيد سمك طبقة الطلاء للالكيد وبذلك يقل معدل عملية افراز السماد وكذلك يوصى بأستخدام الالكيد طويل الزيت لرخص ثمنة وسهولة تحللة لوجود نسبة عالية من الزيت فى التركيب ويعتبر صديق للبيئة ولا يؤثر عليها.