Characterization of maleated vegetable oils for insulation purposes and agricultural applications

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Abstract: Two maleated vegetable oils based on castor and soybean oils (COMA & SOMA) in addition to aliphatic polyester based on propylene glycol and succinic acid (PPS) were prepared and characterized by means of infrared (IR), nuclear magnetic resonance (^{1}H NMR) and thermogravimetric analysis (TGA). TGA data indicates that SOMA is more thermally stable than COMA and PPS. The electrical properties of the prepared materials were investigated through the permittivity (ε'), the dielectric loss (ε'') and the electrical conductivity (σ) in a frequency range 10^{-1}—10^{+7} Hz. The obtained data were found to follow the trend COMA > SOMA > PPS. The relaxation mechanisms of such systems were also studied through the dielectric modulus *M*', including the real and imaginary parts (*M*' and *M*''). The effect of the prepared materials on the hydrophysical properties of the sandy soil when supplemented with different concentrations (1—5 wt %) was studied by bulk density, total porosity, water holding capacity, field capacity, hydraulic conductivity and water stability aggregates. These hydrophysical properties were improved by increasing the concentration of the added materials, which can be explained in terms of the penetration resistance in relation to friction and cohesion forces. These improvements in the hydrophysical properties of the sandy soil render it suitable for plant growth.

Keywords: maleated vegetable oils, thermal properties, electrical properties, agricultural applications.

Charakterystyka olejów roślinnych modyfikowanych bezwodnikiem maleinowym, przeznaczonych do zastosowań izolacyjnych i rolniczych

Streszczenie: W wyniku modyfikacji olejów roślinnych bezwodnikiem maleinowym otrzymano dwie pochodne: oleju sojowego — SOMA i rycynowego — COMA. Ponadto w reakcji kwasu bursztynowego z glikolem propylenowym wytworzono alifatyczny poliester—PPS. Syntezowane materiały charakteryzowano z wykorzystaniem metod spektroskopii w podczerwieni (FT-IR), magnetycznego rezonansu jądrowego (^{1}H NMR) oraz analizy termograwimetrycznej (TGA). Wykazano, że SOMA jest stabilniejszy termicznie niż COMA i PPS. Właściwości elektryczne oceniano na podstawie przenikalności (ε'), strat dielektrycznych (ε„) i przewodności elektrycznej σ_{ac}, wyznaczanych w zakresie częstotliwości 10^{-1}—10^7 Hz, a także wartości modułu dielektrycznego (*M*). Najlepsze właściwości elektryczne wykazywała pochodna oleju rycynowego a najgorsze — alifatyczny poliester. Badano wpływ dodatku (1—5 % mas.) otrzymanych materiałów na właściwości hydrofizyczne gleb piaszczystych. Oceniano gęstość nasypową, porowatość całkowitą, zdolność do gromadzenia wody, pojemność wodną, współczynnik filtracji oraz wodoodporność agregatów glebowych. Oznaczane właściwości hydrofizyczne gleby polepszały się wraz ze wzrostem zawartości dodatku, co można tłumaczyć zmieniającymi się siłami kohezji i frykcji. Taka poprawa jest korzystna dla rozwoju systemu korzeniowego roślin.

Słowa kluczowe: oleje roślinne modyfikowane bezwodnikiem maleinowym, właściwości termiczne, właściwości elektryczne, zastosowania rolnicze.

Biobased materials have attracted much attention because of their sustainability and environmental concerns [1, 2]. Recently, renewable materials synthesized from plant products have shown great promise as a replacement for petroleum-based materials [3]. Thermosetting resins [4], biodegradable polymers [5], biocomposites [6] and polyurethane dispersions [7, 8] from acylated epoxidized soybean oil, soy protein and vegetable oil-based polyols have been successfully

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synthesized by free radical, ring opening and addition polymerizations.

Castor oil was subjected to many familiar organic reactions to form useful derivatives that can undergo radical or condensation polymerization reactions. A recent work reported the synthesis of biodegradable and cross-linkable poly (castor oil fumarate)/poly (propylene fumarate) composite adhesive as a potential injectable and *in situ* cross linkable polyester resins for orthopedic applications [9]. In another study, the preparation of biodegradable foam plastics based on castor oil was reported [10]. Can *et al*. [11] studied the mechanical properties of soybean and castor oil based on thermosetting polymers. They prepared maleic anhydride modified soybean and castor oil based monomers *via* the maleination of the alcoholysis products of the oils with various polyols. The polymers prepared from castor oil exhibited significant improvement in modulus strength and glass transition temperature when compared with polymers based on soybean oil.

A series of measurements were carried out to study the dielectric properties of ten edible oils including soybean oil in the frequency range 100 Hz—1 MHz, showing a decrease in the dielectric loss ε'' up to 13.2 kHz and then an increase at higher frequencies [12]. The dielectric properties, as well as the electrical conductivity measurements of castor oil [13] and castor oil esterified with some acid anhydride, were studied in our previous work [14] at different frequencies 10^2—10^5 Hz to be used for insulation purposes. Sandy soils are often considered as soils with physical properties far from simple. This is particularly the case for sandy soils in the tropics where they are subjected to a cycle of wetting and drying associated with seasonality. In this respect, small changes in composition lead to significant differences in physical properties. One of the major soil characteristics to be taken into account is the size distribution of the sand grains. Fine sand induces greater porosity, water retention and resistance to penetration than coarse sand.

Soil aggregates are groups of soil particles that are bound to each other more strongly than to adjacent particles. Organic matter „glues”, produced when soil biota break down dead roots and litter, hold the particles together. Aggregate stability refers to the ability of aggregates to resist degradation. The additions of organic matter to the soil enhance the stability of aggregates. Stable aggregates are critical to erosion resistance, water availability, and root growth. Soils with stable aggregates at the surface are more resistant to water erosion than other soils [15].

Polycyclic aromatic hydrocarbons (PAH) are organic compounds formed by the incomplete combustion of organic matter, or as a result of anthropogenic activities (*e.g.* coal tar, petroleum or wood-preserving industries), some of which are classified as human carcinogens [16, 17].

Vegetable oil has the ability to extract (PAHs) from contaminated sandy soil for remediation purposes, with some of the oil remaining in the soil.

The effect of rapeseed oil (0.0, 0.1 and 1 wt %) on the degradation of polycyclic aromatic hydrocarbons (PAH) by *Rhodococcus wratislaviensis* was studied in soils artificially contaminated with phenanthrene, anthracene, pyrene and benzo(a)pyrene (50 mg) each, for 49 days at 30 °C [18].

In this work, a comparative study was performed on the thermal, electrical properties and relaxation mechanisms of the two prepared maleate vegetable oils (based on castor oil and soybean oil) and aliphatic polyester based on propylene glycol and succinic acid. Also, the effect of different concentrations (1—5 wt %) of the prepared COMA, SOMA and PPS on the hydrophysical properties of sandy soil was investigated aiming to improve such properties rather than extraction of PAH from the soil to render them suitable for plant growth.

EXPERIMENTAL PART

Materials

The materials used in this study (castor oil and soybean oil) were obtained from ABCO Chemie, Gillingham, England. Maleic anhydride, succinic acid, 1,2-propanediol, hydroquinone and *N*,*N*-dimethyl benzylamine were obtained from Merck, Darmstadt, Germany. Tetrabutyl titanate, $Ti(OBu)_4$, was used as the transesterification catalyst, reagent grade was obtained from Sigma-Aldrich, Steinheim, Germany. Sandy soil was obtained from El-Nubaria, Egypt.

Preparation of maleated castor oil (COMA)

COMA was prepared by the reaction of castor oil (CO) with maleic anhydride (MA) at a mole ratio 1:3. Castor oil 92.5 g, maleic anhydride 29.4 g and 0.012 wt % hydroquinone were placed in a three-necked, round-bottom flask equipped with a glass stirrer and a thermometer. The reaction proceeded with continuous stirring in an oil bath and the mixture was heated to 90 °C. When the maleic anhydride melted, 0.12 wt % of *N*,*N*-dimethyl benzylamine was added and the reaction temperature was stabilized at 98 °C. The mixture was agitated at this temperature for 5 h. The obtained product was a light yellow, viscous liquid at room temperature.

Synthesis of maleated soybean oil (SOMA)

Soybean oil (87 g, 0.1 mol, assuming an average molecular weight 870) was placed in a flask under nitrogen fitted with a condenser, magnetic stirrer, and thermometer. The soybean oil/maleic anhydride ratio was 1:2.4 mol.

Equal amounts of maleic anhydride (7.84 g, 0.08 mol) were added every hour for three hours. The mixture was heated to 180 °C for the first hour then the temperature was raised 20 °C every hour. At the end of the reaction, the mixture was stirred for another 1 h at 230 °C and allowed to cool to room temperature.

Synthesis of poly(propylene succinate) (PPS)

Aliphatic polyester poly(propylene succinate) was synthesized in bulk by the transesterification reaction in the presence of the catalyst $Ti(OBu)_4$. A three necked flask equipped with a condenser, nitrogen inlet tube and magnetic stirrer was charged with (one mol) of succinic acid and (1.1 mol) of 1,2-propanediol and the catalyst. The reaction mixture was purged with nitrogen and it was then heated at 170 °C for 5 hours. The water formed during the reaction was distilled off. In the second step of the reaction, the mixture was subsequently heated to a final temperature 240 °C for another one hour in order to distill off the traces of water. The obtained polyester had a yellowish color.

Methods of testing

— Infrared spectra were recorded on a Jasco FT/IR 300 E Fourier transform infrared spectrometer (Tokyo, Japan).

— ^{1}H NMR spectrum was run at a Jeol-Ex-270 NMR spectrometer (Tokyo, Japan).

— TGA was performed using a Perkin-Elmer, TGA 7 (USA) instrument. The rate of heating was 10 °C/min up to 700 °C under a nitrogen atmosphere.

— The dielectric permittivity ε', dielectric loss ε'', and conductivity σ (obtained from direct resistant measurements) were measured over a frequency range 0.1 Hz—5 MHz at room temperature (~30 °C), using an impedance analyzer (Schlumberger Solartron 1260), an electrometer, amplifier, and measuring cell as described before [19]. The error in ε' and ε'' amounts to 1 % and 3 %, respectively. The temperature of samples was controlled by a temperature regulator with a Pt 100 sensor to avoid moisture, thereafter the samples were stored in desiccators in the presence of silica gel. The sample was transferred to the measuring cell and left with P_2O_2 until the measurements were carried out.

— The hydrophysical properties of the sandy soil were studied through the bulk density, total porosity, water holding capacity, field capacity, hydraulic conductivity and water stability aggregates according to the methods described by Klute [20].

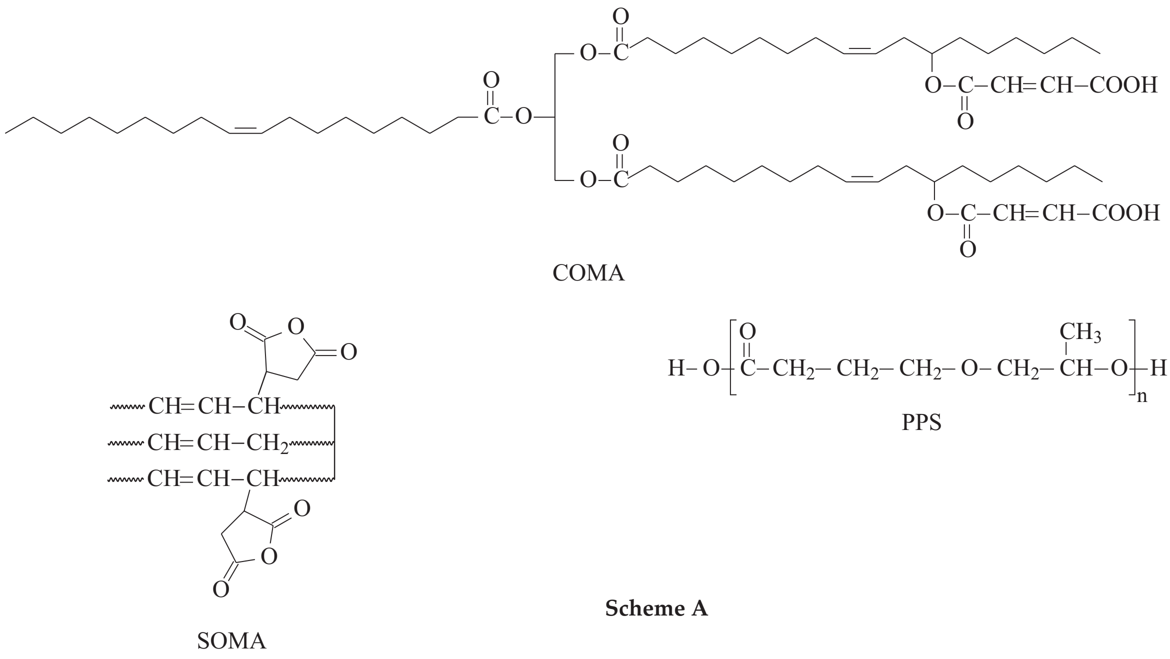
RESULTS AND DISCUSSION

FT-IR and ^{1}H NMR spectroscopies

The chemical structure of the maleated castor oil (COMA), maleated soybean oil (SOMA) and poly(propylene succinate) (PPS) are shown in Scheme A as characterized by means of FT-IR and NMR spectroscopies.

FT-IR spectra of castor oil and COMA are compared in Fig. 1. For castor oil, the broad peak at 3434 cm^{-1} can be ascribed to the -OH absorption, while that at 1640 cm^{-1} can be assigned to the -HC=CH- double bond stretching frequency. When castor oil reacts with maleic anhydride (COMA), the -OH peak was evidently decreased, while the double bond peak was enhanced. No peaks corresponding to cyclic anhydride at 1779 cm^{-1} and 1849 cm^{-1} were found, indicating that almost all of the maleic anhydride was consumed in the reaction with castor oil.

Figure 2 represents the ^{1}H NMR spectra of castor oil and COMA. From this figure, it is clear that when castor oil was converted to COMA, the peak related with the hydroxyl (CH-OH) at 3.61 ppm nearly disappeared, while a new peak represents the methylene protons connected to maleate groups (CH-O-CO) at 5.03 ppm. The two well-defined doublets at 6.2—6.5 ppm indicate the two olefinic protons of maleic anhydride moiety compared with the NMR spectrum of castor oil, while the peak



Scheme A

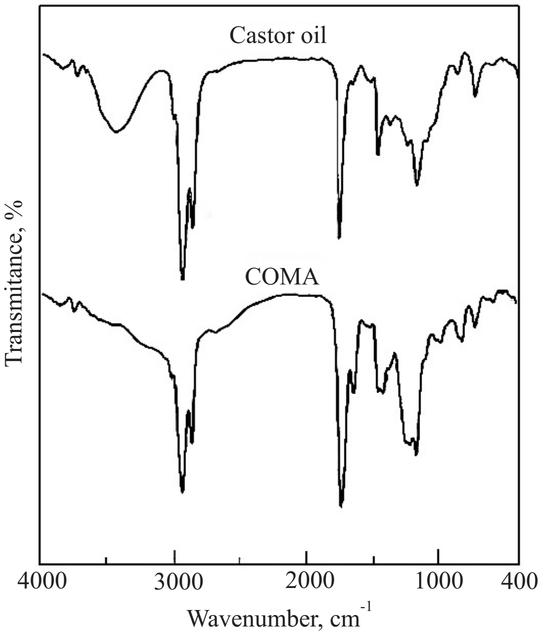


Fig. 1. FT-IR spectra of castor oil and COMA

at 7.1 ppm is assigned to the unreacted maleic anhydride residue.

The FT-IR spectrum of soybean and SOMA are shown in Fig. 3. The characteristic peaks of soybean oil appeared at 3007, 2925 and 2856 cm^{-1} due to the stretching frequency of CH, CH_2 and CH_3 and the peaks at 1650, 1458 cm^{-1} (correspond to -CH=CH-). The peak of the carbonyl group of soybean oil at 1745 cm^{-1} is split into two peaks (1739 and 1713 cm^{-1}) and the peak at 960 cm^{-1} may be attributed to the presence of acid anhydride. Also, the absence of a carboxylic acid band indicates that the anhydride was intact.

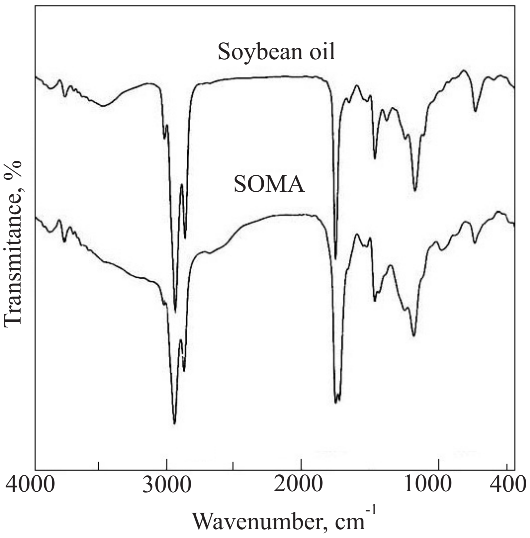


Fig. 3. FT-IR spectra of soybean oil and SOMA

The ^{1}H NMR spectra of soybean and SOMA (Fig. 4) showed a similar group of signals at δ: 0.86 (CH_3-); 1.2 (-CH_2-); 2.3 [-CH_2-C(=O)-O]; 2.7 (-CH=CH-$\underline{CH_2}$-CH=CH-); 4.2 [-O-CH_2-$\underline{CH}$(O)-]; 4.1 [-O-$\underline{CH_2}$-CH(O-)-CH_2-O-]; 5.2 (-HC=CH-). The methylene signals of succinate hydrogen cannot be determined by ^{1}H NMR due to its coincidence with that of the soybean methylene protons.

The FT-IR spectrum of poly(propylene succinate) (PPS) (Fig. 5) indicates the presence of an intense, strong band at 1737 cm^{-1} characteristic of the stretching frequency of acid and ester carbonyl groups C=O and the band of C-O-C appears at 1166 cm^{-1}. The broad band appearing at 3440 cm^{-1} stands for OH groups, and the bands at

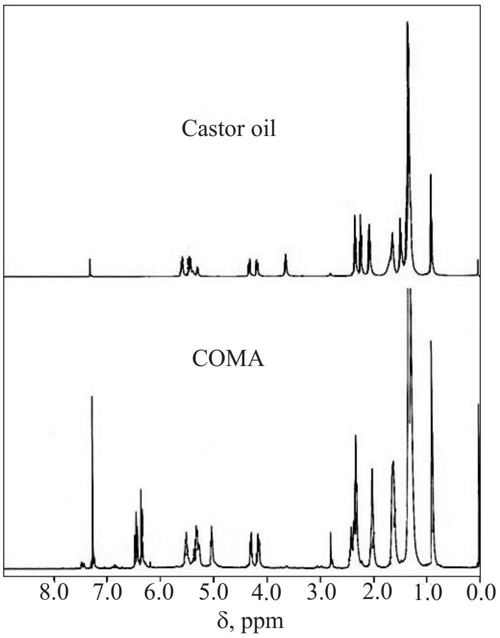


Fig. 2. ^{1}H NMR spectra of castor oil and COMA

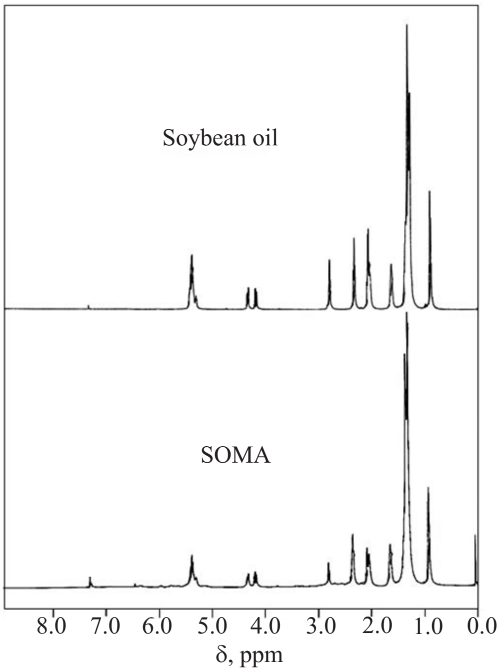


Fig. 4. ^{1}H NMR spectra of soybean oil and SOMA

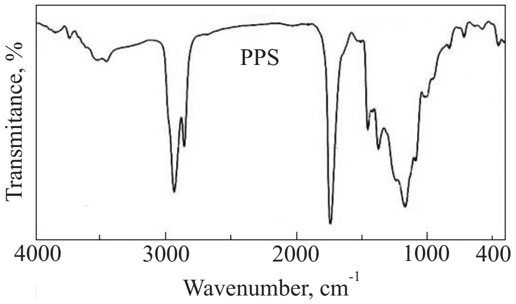


Fig. 5. FT-IR spectra of PPS

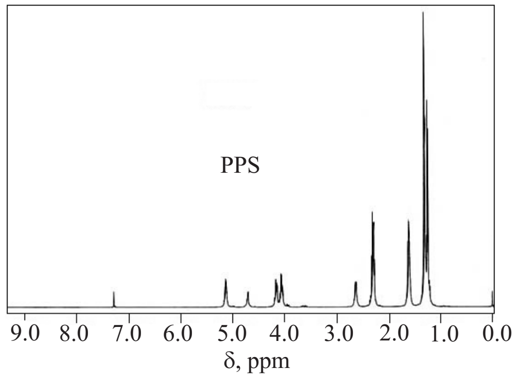


Fig. 6. ^{1}H NMR spectra of PPS

2860—2932 cm^{-1} are due to the stretching frequency of CH_2 and CH_3.

The ^{1}H NMR spectra of PPS (Fig. 6) showed a group of signals characteristic for the methyl, methylene, and methine proton peaks of 1,2-propane diol that appeared at 1.2, 4.1 and 5.1 ppm, respectively. The signals at 2.2 ppm may be attributed to the methylene protons of succinic acid.

TGA analysis

Thermal degradation of COMA, SOMA and PPS was studied by determining its mass loss during heating. Figure 7 shows the TGA thermogram of COMA, SOMA and PPS. It was observed that the maleated soybean oil (SOMA) shows an increase in thermal stability compared to maleated castor oil (COMA) and PPS. The decomposition of all samples show only one degradation step, starting above 160 °C. In order to compare the relative thermal stability of the prepared samples, the temperatures at mass losses of 10, 25, 50 and 75 % are presented in Table 1. It is clear that the temperatures at these mass losses are shifted towards higher values in the case of SOMA.

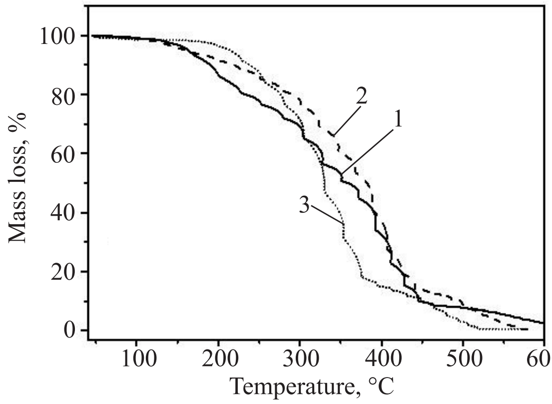


Fig. 7. TGA curves of COMA, SOMA and PPS

T a b l e 1. TGA data of COMA, SOMA and PPS

Samples	Temperature at characteristic mass loss, °C			
	$T_{10\,\%}$	$T_{25\,\%}$	$T_{50\,\%}$	$T_{75\,\%}$
COMA	190	268	358	412
SOMA	219	309	382	417
PPS	238	288	330	369

Dielectric measurements

The permittivity ε′ and dielectric loss ε″ of COMA, SOMA and PPS were measured over a frequency range (0.1 Hz—1 MHz) at room temperature (30 °C). The obtained results are shown graphically in Fig. 8. The data for ε′ and ε″ increases over the whole frequency range, following the trend COMA > SOMA > PPS. The higher values of ε″ given at lower frequencies could be attributed to the conductivity contribution, which is more pronounced in the case of COMA. The broadness of the absorption cur-

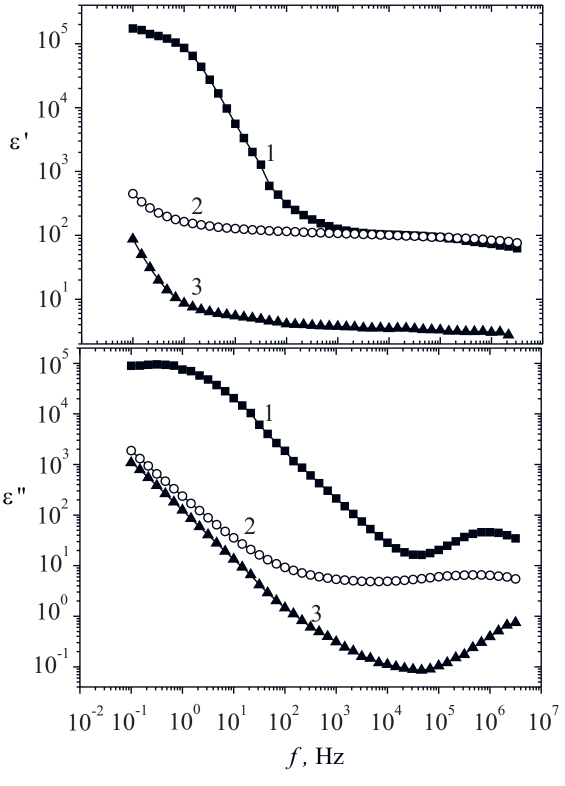


Fig. 8. The permittivity ε′ and dielectric loss ε″ as a function of frequency f at room temperature (~30 °C) for: 1 — COMA, 2 — SOMA, 3 — PPS

ves relating to ε'' and log *f* indicates that more than one relaxation mechanism is present.

The relaxation mechanism of the systems can be explained by the electric modulus $M^* = M' + M''$ where M' and M'' are the real and imaginary parts respectively of the electric modulus M^* and equals to $[M' = \varepsilon'/(\varepsilon')^2 + (\varepsilon'')^2$ and $M'' = \varepsilon''/(\varepsilon')^2 + (\varepsilon'')^2]$.

The main advantage of the above presentation is that the contribution of the electrode polarization effects is negligible, allowing evolution processes at the lower frequency range of dielectric spectra. These processes are mainly governed by either Maxwell-Wagner Sillar (MWS) effects or the total conductivity of the measured samples, appearing as a steady increase in the ε'' values as it is transformed into a loss peak in the modulus presentation, or both.

M' and *M''* were calculated for the three investigated systems and illustrated graphically in Fig. 9. To specify the relaxation mechanisms of such systems, the data of *M''* versus the different frequencies were analyzed using Havriliak-Negami and Frohlich functions [21] as shown in Fig. 10. Three relaxation mechanisms are detected. The first relaxation mechanism, in the lower frequency range, which is fitted by the Havriliak-Negami function, could ascribe the conductivity relaxation. The second relaxation mechanism, which is fitted by the Frohlich function, could be attributed to the Maxwell-Wagner effect [22] and the difference in conductivities and permittivities of the constituents of the investigated systems. The third relaxation process, in the higher frequency range, is found

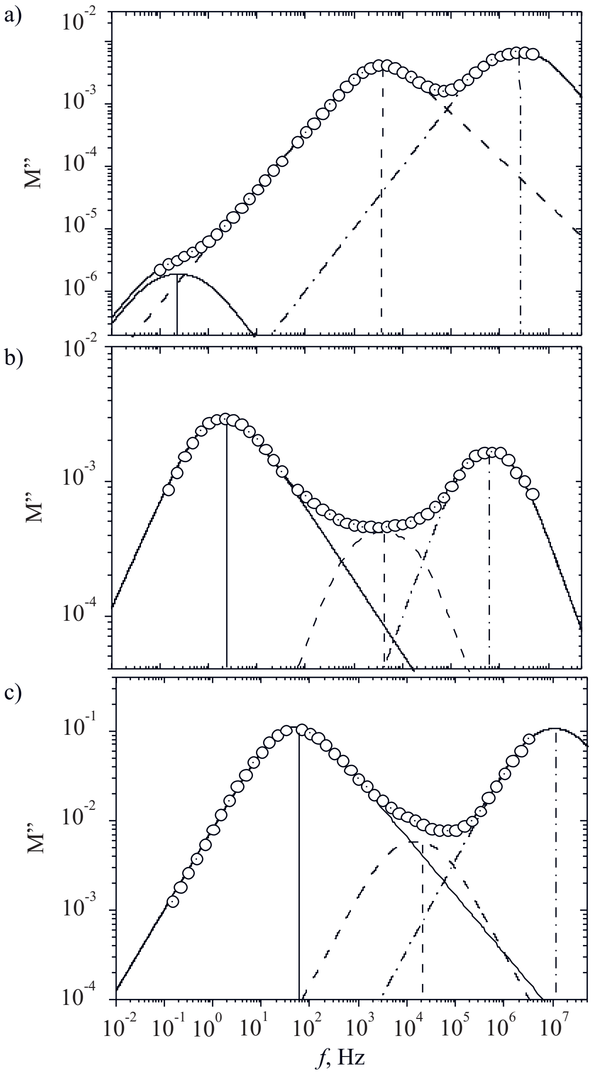


Fig. 10. Imaginary part of electric modulus *M''* as a function of frequency *f* at room temperature (~30 °C) for: 1 — COMA, 2 — SOMA, 3 — PPS

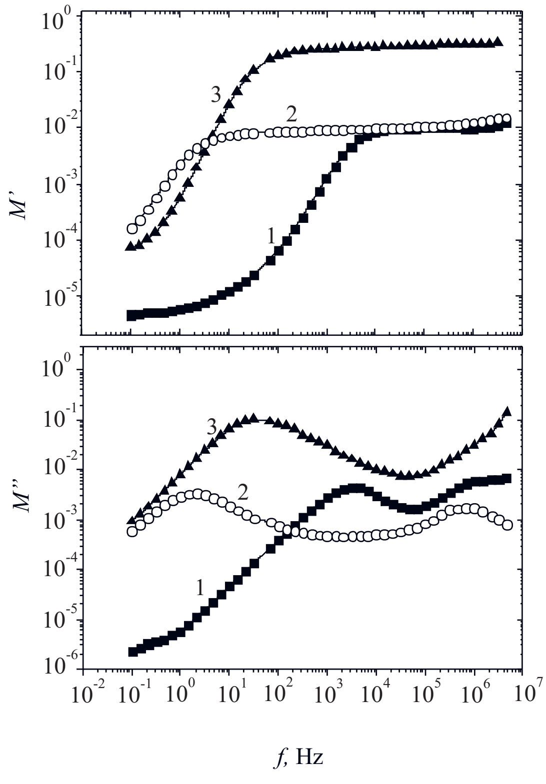


Fig. 9. The dielectric modulus *M* (real *M'* and imaginary *M''*) as a function of frequency *f* at room temperature (~30 °C) for: 1 — COMA, 2 — SOMA, 3 — PPS

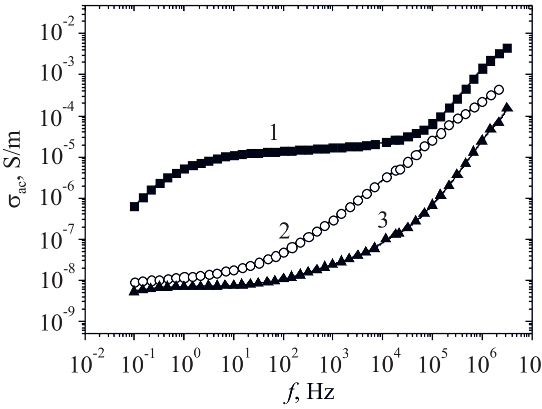


Fig. 11. Electrical conductivity σ_{ac} (s/m) as a function of frequency *f* (Hz) at room temperature (~ 30 °C) for: 1 — COMA, 2 — SOMA, 3 — PPS

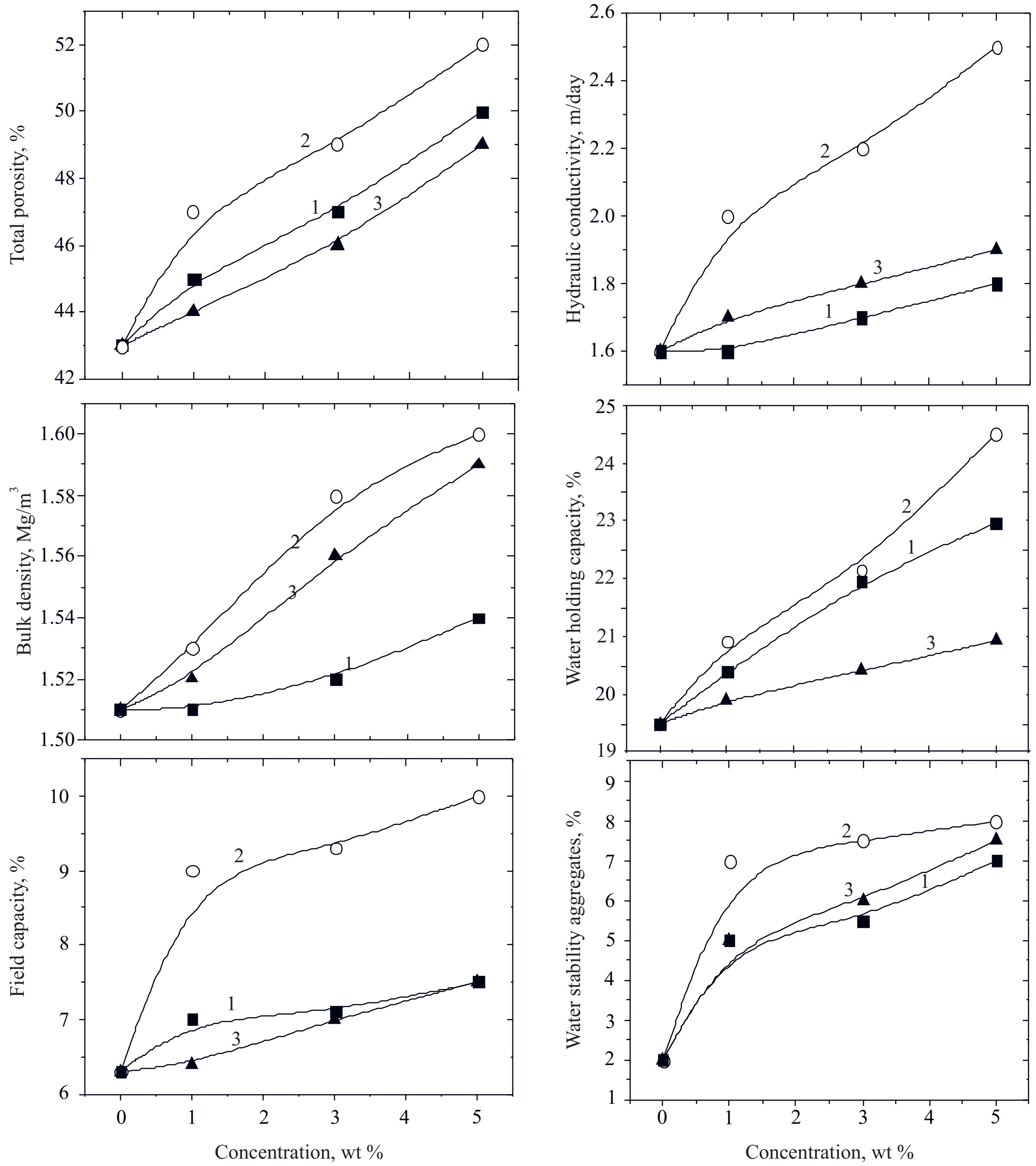


Fig. 12. Physical properties of sandy soil by the addition of different concentrations of: 1 — COMA, 2 — SOMA, 3 — PPS

to be fitted by the Frohlich function and expected to ascribe segmental orientation. The relaxation time $\tau_3 = 1/2\pi f_m$ given for such a process is found to follow the trend SOMA > PPS > COMA by the value $6.3 \cdot 10^{-8}$ s, $2.6 \cdot 10^{-7}$ s and $1.4 \cdot 10^{-8}$ s for COMA, SOMA, and PPS, respectively. The highest value of τ_3, obtained in the case of SOMA, could be attributed to the presence of succinic anhydride attached to the main chain.

Electrical conductivity measurements

The variation of electrical conductivity σ_{ac} for the three investigated materials as a function of frequency shown in Fig. 11 follows the trend COMA > SOMA > PPS. σ usually takes the opposite trend when compared with the data of ε' given in Fig. 8. The d—c conductivity (σ_{dc}) was calculated for the investigated materials and gave the values 10^{-5}, 10^{-8} and $8 \cdot 10^{-9}$ Sm^{-1} for COMA, SOMA and PPS, respectively. From these data, it is seen that σ_{dc} for SOMA and PPS lies in the moderate range of conductivity, which indicates that both materials could be used for antistatic applications [23].

Effect of COMA, SOMA, and PPS on sandy soil properties

Sandy soils are often considered as soils with physical properties that are easily defined. However, they are far

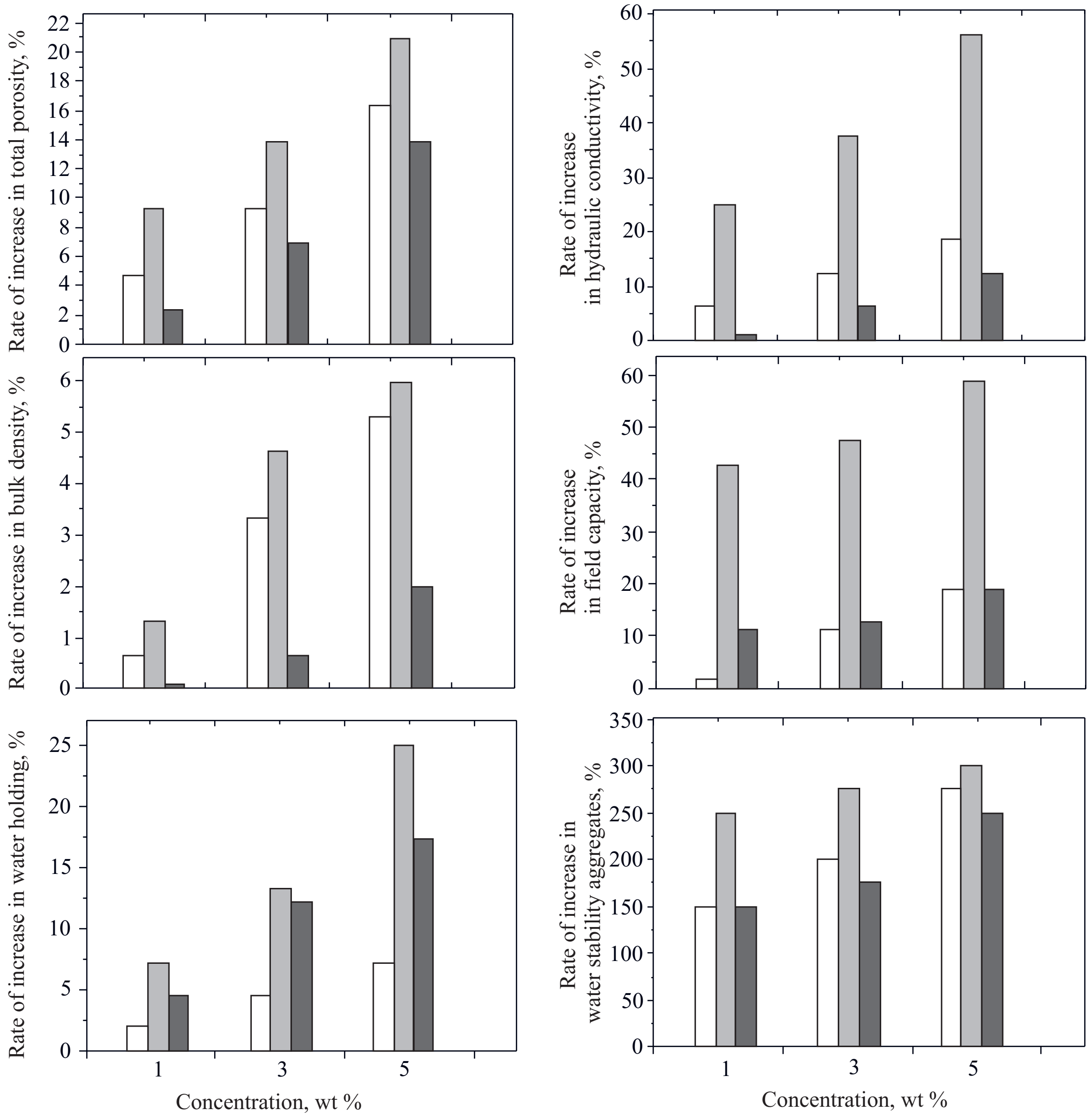


Fig. 13. Rate of increase of physical parameters of sandy soil by the addition of different concentrations of: ☐ — COMA, ▒ — SOMA, ■ — PPS

from being simple. These properties, as mentioned, are bulk density, total porosity, water holding capacity, field capacity, hydraulic conductivity and water stability of the aggregates. The presented data for these properties are given in Table 2. The given data are the mean values for at least three readings.

T a b l e 2. Physical characteristics of the control soil

Physical parameters	Value
Bulk density D_b, Mg/m^3	1.51
Total porosity Q, %	43.0
Water holding capacity, %	19.6
Field capacity, %	6.3
Hydraulic conductivity K_s, m/day	1.6
Water stability aggregates, %	2.0

The values of bulk density and total porosity given in Table 2 are consistent with those obtained earlier for sandy soils in the tropics [24, 25]. These values show a large range of porosities with bulk densities (D_b) from 33 % ($D_b = 1.789$ cm^{-3}) to 47 % ($D_b = 1.789$ cm^{-3}). The large range of porosity is related to the small cohesion forces between elementary particles thus enabling the formation of a large range of assemblages from very loose to very compact. The porosity in sandy soils is usually smaller than clayey and silty soils. The saturated hydraulic conductivity (K_s) is also comparable with those found in literature by Prevedello *et al*. 1995 [26] where it varies within a range of values covering several orders of magnitudes ($10^{-7} < K_s < 10^{-3}$). K_s usually varies according to the development of macroporosity. As a consequence, K_s variation is more closely related to macroporosity development than soil texture. Most studies try to relate K_s to the part of the macroporosity that is called effective porosity (Q_e) and defined as total porosity Q minus the water content. The very small inter-particle cohesion in the sandy soil results in very small aggregate stabilities, which in turn makes it highly sensitive to surface casting.

In order to improve the physical properties of sandy soil to make it more suitable for plant growth, COMA, SOMA and PPS were added at various concentrations (1—5 wt %) to the control soil. Such materials have the ability to extract polycyclic aromatic hydrocarbons (PAHs) from the contaminated sandy soil for remediation purposes with some of the oil remaining in the soil. The details of PAHs were discussed before [27].

The physical properties of the sandy soil after the addition of the investigated polyesters are shown graphically in Fig. 12. All parameters were found to improve after increasing the concentration of the materials added to the soil.

The rate of increase in each parameter was calculated versus the concentration of the materials used in Fig. 13. The increase in bulk density and total porosity invariably results in an increase in the penetration resistance with significant consequences for root development as the presence of such materials could act as inter-grain cement, which can lead to very different physical soil properties.

The aggregate stability is the ability of the bonds of the aggregates to resist when exposed to stresses causing their disintegration. The rate of increase in water stability aggregates (Fig. 13), ranging from 250 to 300 %, indicates that the addition of the investigated materials to the soil enhances such stability, which is critical to erosion resistance, water availability and root growth.

The remarkable increase in these physical parameters after the addition of the investigated materials to the sandy soil is considered to be evidence for improving the hydro physical properties of the soil to make them more suitable for plant growth. Moreover, it is of great interest to find that the rate of increase is higher in the case of SOMA when compared with those for COMA and PPS.

CONCLUSIONS

Two maleated vegetable oils based on castor oil, soybean oil and PPS were prepared and characterized by means of infrared (IR), nuclear magnetic resonance (^{1}H NMR) and thermogravimetric analysis (TGA). SOMA was found to be more thermally stable than COMA and PPS. The dielectric permittivity ε′, dielectric loss ε′′ in addition to the electrical conductivity σ were studied over a frequency range (10^{-1}—10^6 Hz), showing an increase in the order COMA > SOMA > PPS. The moderate range of d—c conductivity noted in the case of SOMA and PPS indicated that both materials could be used for antistatic applications. The relaxation times of the investigated materials, which were obtained from the dielectric modulus data, were found to follow the order SOMA > PPS > COMA. The highest value of relaxation time obtained in the case of SOMA could be attributed to the presence of succinic anhydride attached to the main chain. The hydrophysical properties of sandy soil were improved by the addition of the investigated materials and increase by higher concentrations of such materials following the trend SOMA > PPS > COMA. The resulting improvement of the hydrophysical properties of the sandy soil make it suitable for plant growth.

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