

Characterization of blend and composites for agricultural use at arid and semiarid soils

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ABSTRACT

The use of polymers as soil-stabilizer additive has expanded significantly in agricultural use to control soil degradation, desertification and to improve arid and semi arid soils. This research was conducted to determine the effects of different synthetic polymers and biopolymers at low concentration (0.03 %-1%) at arid and semi arid soil of North Africa. Polystyrene (PS), Polyacrylamide (PAM); Cellulose and the mixture of PAM and PS were characterized by viscosity, infrared spectroscopy, X-ray Diffractometry, thermal analysis (TG and DSC) and Scanning electron micrographs (SEM). The results showed that composites from polymers: PAM and PS with Cellulose in soil, could improve better, soil physical proprieties and augment water retention at arid soils compared with application of any other polymers at the same concentrations. This work can help to improve the productivity of arid and semi arid soils by using low concentration of biopolymers from plant fibers and polymers from synthetic plastics compounds or the wastes of plastic industry, aiming the increase of water holding capacity and also to improve physical properties of soils by binding soils particles together. The reduction of the water loss; also to make valuable wastes of plastic industry and renewable organic fibers to protect environment.

Key words: Composites, Polymer, Polyacrylamide, Polystyrene,
Arid and semi arid Regions, Cellulose, Biopolymers, Wastes.

Abbreviations: (PAM) - PolyAcrylamide; (Cell)- Cellulose;
(PS)- Polystyrene; (V/V)- volume to volume.

INTRODUCTION

Arid and semi arid soil are characterized by low erratic rainfall and long periodic droughts, high evaporation level. Soil degradation threatens water quality and agricultural productivity through the loss of valuable top soil. Polymers and biopolymers have long been recognized as viable soil conditioners, because they stabilize soil surface structure and pores continuity.

In the past decade water soluble polymers was identified as a high effective erosion preventing

and infiltration enhancing polymers, (Lentz *et al*, 1994).

Polyacrylamide was effective in enhancing the stability of soil aggregates (Steven Green *et al*, 2004) and increase soil infiltration in some areas especially in sandy loam soils (Steven Green *et al*, 2000; Hussein and Thomas, 2006). Polyacrylamide is a long-chain synthetic polymer that acts as a strengthening agent, binding soil particle together and holding soils in place, but Polyacrylamide alone not remediate poor soil Structure, (Cook and Nelson, 1986).

Acid - hydrolyzed Cellulose micro fibrils alternative to Polyacrylamide for soil stabilization was tested and show promises (Orts et al, 2007). The general objective of this study was to produce a series of low costs polymers and biopolymers to quantify their effectiveness on soil retain water, decrease evaporation; improve soil aggregate stability and other structural properties.

The present work tends to make valuable products of plastics wastes (Polystyrene) and synthetic compounds (Polyacrylamide) with Cellulose and soil as a composite materials to retain water and humidity for crops and plants at arid and semi arid regions.

The uses of Polyacrylamide-Cellulose blend appears promising for reducing the cost of irrigation at arid and Semi-arid soils, and offer a safe environmentally friendly, inexpensive materials. The environmental advantages of degradable sources may give the Cellulose Alfa-Alfa economic advantages, as a source of polysaccharides to arid soils that generally lack structural polysaccharide; also Cellulose Alfa-Alfa. (*Stipa tenacissima*) is very rich plant in Cellulose fibre, also it resist very well to drought at semi arid regions, in the same time it's annually renewable and its additions would generally be regarded as environmentally beneficial. Polystyrene used as impermeable subsurface layer barrier for water movement during irrigation or

rainfall provide also deep percolation of water.

EXPERIMENTAL

Material and Methods

Styrene (Aldrich 99 %), Acrylamide was supply by (E.MERCK) was purified by crystallisation and recrystallisation in hot methanol; crystals are separated by filtration then desiccated under vacuum at 30 C°.

Also we purified Potassium persulfate as the same manner.

Water used as solvent was bidistilled water at KMnO_4 .

Methanol 215 (Romil-SPS 99.9 %), ethyl benzene (Carlo Erba, chimica Milano), Toluene (BDH chemical LTD)

Acetone (Romil-SA), Potassium persulfate (99.9 %) and Benzoyl peroxide (98 %) (Sigma - Aldrich). All other reagents were used as received.

Extraction of Cellulose from Alfa-Alfa plant

The cellulose was extracted from alpha plant with toluene/ethanol mixture (2/1, V/V) for 6 hours in Soxhlet apparatus and treated with NaOH (1M) for 8 hours at 25°C (Shigemasa *et al*, 1990; Heinz *et al*, 2000).

Table1: Synthetic details of blend and composites (polymers with soil)

Sample N°	Soil type (50g)	Cellulose (%)	Polyacrylamide (%)
1	Arid	0.5g/l (1%)	15 mg/l (0.03%)
2	Arid	0.5g/l (1%)	0 mg /l
3	Arid	0 g/l	15 mg/l (0.03%)
4	Semi Arid	0.5g/l (1%)	15 mg/l (0.03%)
5	Semi Arid	0.5g/l (1%)	0 mg/l
6	Semi Arid	0 g/l	15 mg/l (0.03%)

Table 2: Physical and chemical proprieties of arid soils (0-20cm)

S %	(C + Si) %	CaCO _{3T}	EC (mS/cm)	MO %	CEC (meq/100g Soil)	PH	Soil textural class
74.52	25.48	5.17	0.23	0.50	3.2	7.84	Sandy

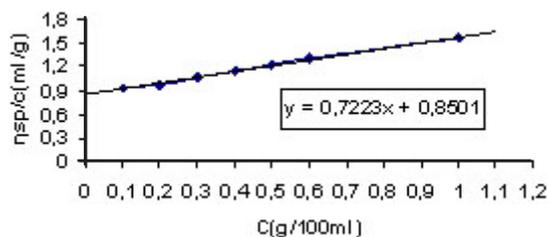


Fig. 1: Reduced η versus concentration of PAM

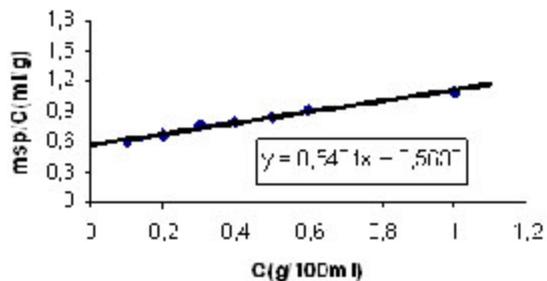


Fig. 2: Reduced η versus concentration of PS

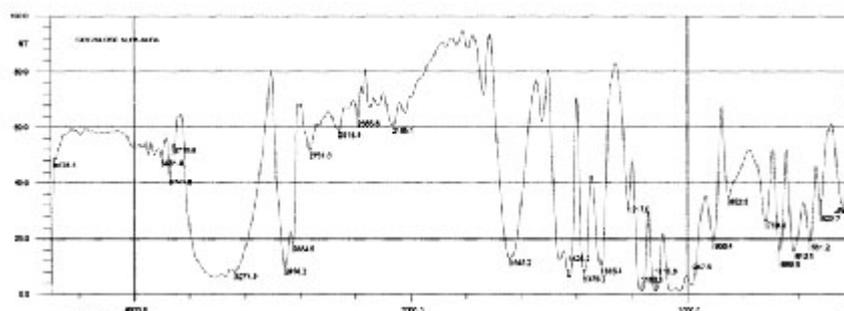


Fig. 3: FTIR spectrum of Cellulose fibres with KBr

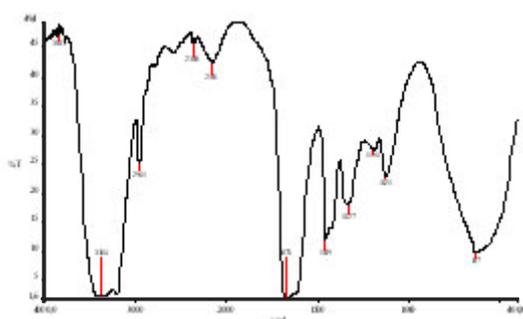


Fig. 4: FTIR spectrum of (PAM) film

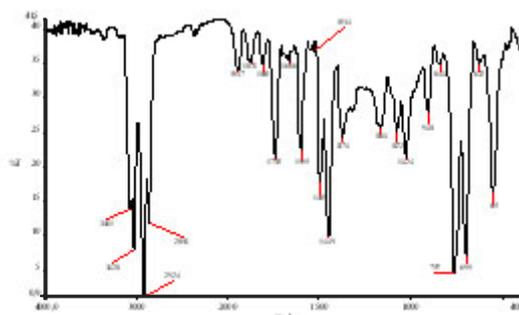


Fig. 5: FTIR spectrum of (PS) film

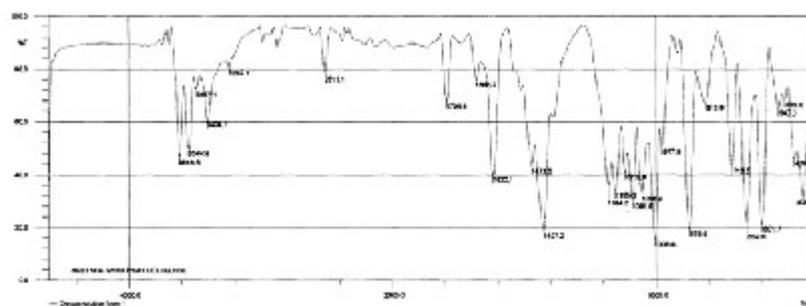


Fig. 6: FTIR spectrum of arid soil with (Polyacrylamide + Cellulose) with KBr

Preparation of Polyacrylamide (PAM)

For the polymerisation reaction we used the method proposed by Schultz (Schultz *et al.*, 1959).

Polyacrylamide was prepared by radical polymerisation in aqueous solution with acrylamide as monomer.

(8g dissolved in 260 ml of bidistilled water), potassium persulfate $K_2S_2O_8$ (0.4g dissolved in 10 ml of distilled water) as initiator, the reaction lasted for 5 hours at constant temperature under inert anhydride nitrogen.

Vigorous stirring ensured homogeneous conditions by RW 20 IKA-WERK motor.

The polymer solution obtained was concentrated by "Rota vapor" (1/3 of initial volume), then it precipitated in methanol (V/V, 1/7) and filtered at Buchner.

The obtained Polyacrylamide was cleaned by dissolved it in bidistilled water then precipitated second time in methanol to eliminate all initiator and monomer traces, then filtered at Buchner, desiccated using inert nitrogen atmosphere (anhydride N_2) under vacuum at 30°C .

Preparation of Polystyrene (PS)

Polystyrene obtained by radical polymerisation at constant temperature 60°C; we used benzoyl peroxide as catalyst and toluene as a solvent:

In reactor contain styrene and toluene solution ($V_{\text{Styrene}}/V_{\text{toluene}}$, 1/3) immersed in thermostat bath at 60°C° we added slowly benzyl peroxide (10^{-3}mol/l dissolved in 10 ml of toluene); the reaction lasted for 8 hours at constant temperature under inert anhydride nitrogen and continuous stirring.

The polymer solution obtained was concentrated to (1/3 of initial volume), then it

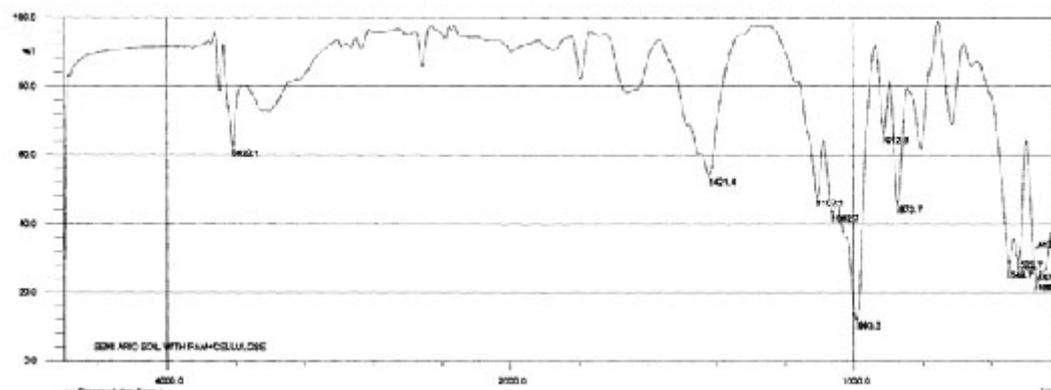


Fig. 7: FTIR spectrum of semiarid soil with (Polyacrylamide + Cellulose) with KBr

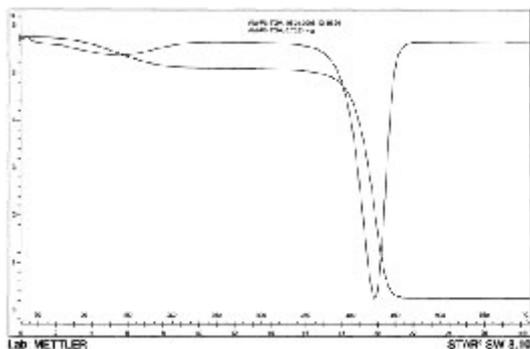


Fig. 8: TGA curve of (PS)

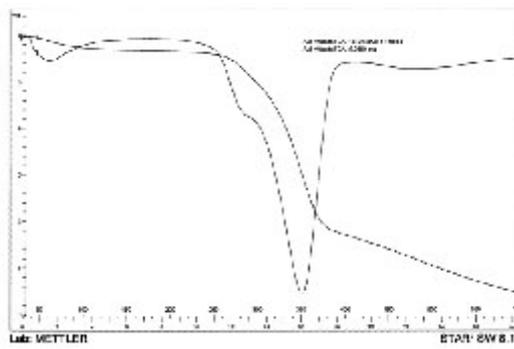


Fig. 9: TGA Cellulose Alfa-Alfa

precipitated in methanol (V/V, 1/7) and filtered at Buchner, The solid obtained rinsed with methanol.

Molecular weight of Polyacrylamide and Polystyrene by Viscosimetry

The viscosity measurements of diluted solutions of polyacrylamide and polystyrene were carried out in a capillary Ubbelohde viscometer 75 in a LAUDA CD 15 bath. The flow times were measured with accuracy of ± 0.01 S. from the efflux time measurement, the specific viscosity (η_{sp}) was calculated at different concentration. The intrinsic viscosity $[\eta]$ was determined by plotting reduced viscosity $[\eta_{sp}/C]$ against the solutions concentrations (C) according to the well known MARK-HOUWINK relation $[\eta] = KM^{\alpha}$, where K and α : are constants for a given polymer/solvent/ temperature system, Viscosity of Polyacrylamide was determined in bidistilled water at 30 C°, the value of $K = 0.65 \cdot 10^{-2}$ and $\alpha = 0.82$ and Viscosity of Polystyrene was determined in Ethyl benzene with $K = 17.6 \cdot 10^{-3}$ and

$\alpha = 0.68$ at 25C ° (Polymer Data Handbook 1999) .

Preparation of blend and composites polymers

Polymer blending provides a relatively facile means of combining the separate desirable properties of different polymers into a single material.

Blends were prepared using solution routes and physical mixing (the latter for DSC measurements only)

Different compositions of Polyacrylamide (PAM) and Cellulose are prepared in wide composition range by solution mixing:

A-(Polyacrylamide-Cellulose) blend was prepared by dispersing Cellulose fibres in Polyacrylamide solution was sonicated for various times between 10 and 15 minutes using ultrasonic, dispersion increased with sonication time and times

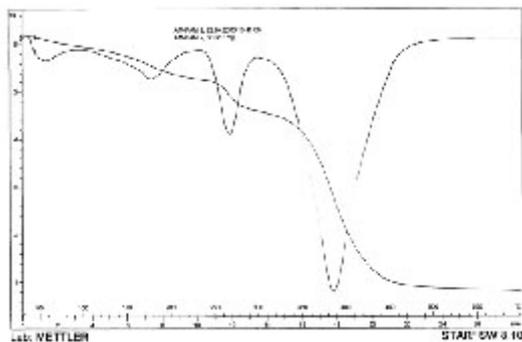


Fig. 10: TGA curve of PAM

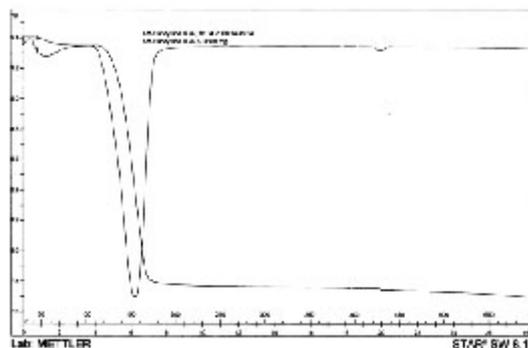


Fig. 11: TGA curve of arid soil

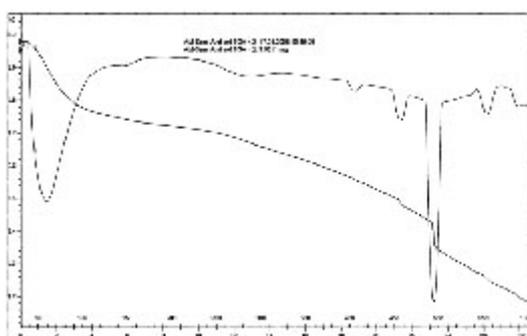


Fig. 12: TGA curve of semiarid soil

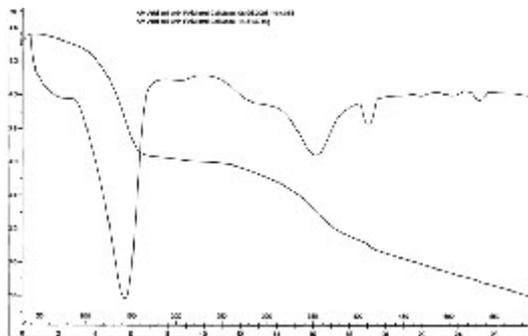


Fig. 13: TGA curve of arid soil with (PAM + Cellulose)

in excess of 15 minutes apparently resulted in the dispersion of cellulose fiber.

B- (Polyacrylamide- Cellulose) Mixture by dispersing Cellulose fibre in acetone solution then we added Polyacrylamide solution with continuous stirring.

C- Simple mixing of Polyacrylamide solution and Cellulose with continuous stirring.

We prepared different blend of Polyacrylamide and Cellulose, using (0 mg/L -20 mg/L) of Polyacrylamide concentration and Cellulose concentration from (0 g /L - 0.5 g/L).

Preparation of Polystyrene sub layer

Polystyrene film was prepared using the

same method used by IKEJIMA *et al.*, 2000; (9) Olabarrieta *et al.*, 2001). We used the casting method (wet method) to prepare Polystyrene film; the thickness of polystyrene film can be controlled by polymer concentration:

We prepared 2- 4 % (w/w) of Polystyrene film by dissolving 5-10 g of polystyrene in 250 ml of toluene under continuous stirring (300 rpm); after filtering the polymer was cast at temperature between 50-60°C then removed from the mould and stocked at 23 ± 2 °C.

Thickness of sub layer film

The thickness of sub layer film was measured at 3 different points with 0.1mm precision using micrometer (Twing -Albert instrument Co., Philadelphia, USA).

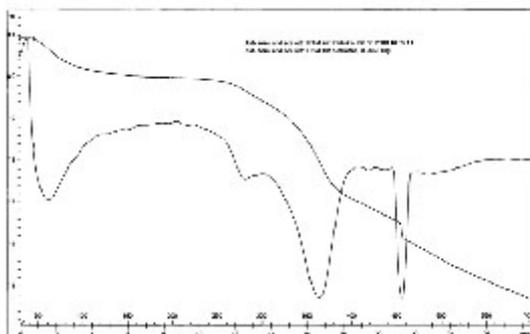


Fig. 14: TGA curve of semiarid soil with (PAM+ Cellulose)

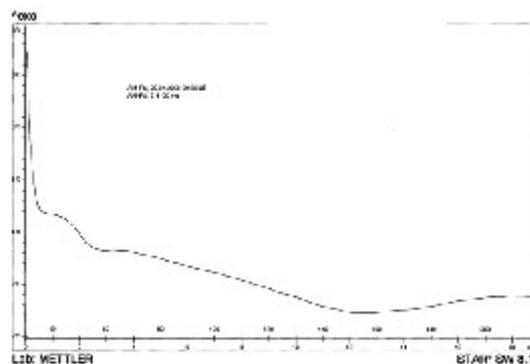


Fig. 15: DSC Polystyrene (PS)

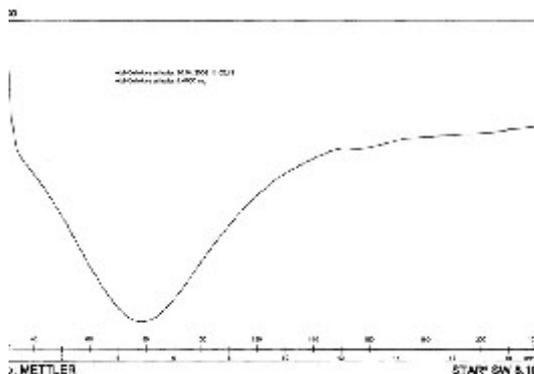


Fig. 16: DSC Cellulose Alfa-Alfa

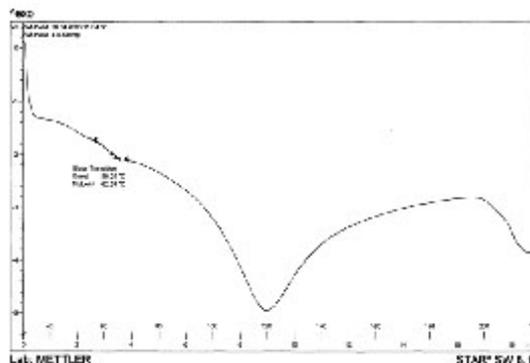


Fig. 17: DSC curves of PA

Soil Specimen preparation

Two soils from Algeria were chosen for this study: arid soil from Biskra (Ain Benoui) and semi arid soil from Batna (Merouana) samples of the soils were taken from the Surface layer (0-20 cm) and were analyzed using standard analytical methods (Klute, 1986; page *et al.*, 1982), selected physical and chemical proprieties of the soils are given in Table 2.

Specimen treated with different biopolymers and polymers Mixtures concentrations. We sprayed Polyacrylamide-Cellulose blend and composite at arid and semi arid soil surface.

Water-retaining property

50g of arid and semiarid soil treated with a different amount of polymers (Polyacrylamide and Cellulose), was put in plastic container (6 cm in diameter, 7 cm in depth) similar to Polystyrene sub-layer to avoid loss of water by deep percolation. Then 20 ml tap water was poured into the container. All container were placed on simulator of temperature and wind at 30°C and 1.2 bars during day, and placed on tables in a constant air-temperature Room (25°C, air relative humidity 20 %) during night, to simulate desert conditions, the water content of each soil sample was determined by taking a small sample from the container every 24 h, oven-drying at 105°C° for 24 h and determining weight loss:

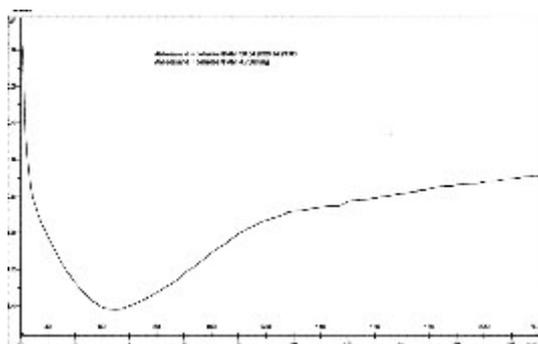
$$\text{Water content (\%)} = (\text{wet soil weight} - \text{dry soil weight}) / \text{wet soil weight} \cdot 100$$


Fig. 18: DSC semiarid soil with (PAM + Cellulose)

Characterisation

Polymers and composites were characterized by elemental analysis: IR spectroscopy, thermal analysis, scanning electron micrographs (SEM), and X-ray diffractometry (XRD).

Elemental analysis

To determine the organic, mineral and dry matter of Alfa-Alfa plant we used the elemental analysis.

FTIR analysis

FITIR spectra were recorded on Perkin-Elmer- Paragon 500 FT- IR spectrophotometer in the range 4000 - 400 cm⁻¹. Using thin film by solution casting via air evaporation and KBr, the thin films were prepared by casting polymers solutions on glass plates.

Thermal analysis

Thermal analysis was carried out with Mettler TATC 11 thermal analyser.

Both thermo gravimetric analysis (TGA) and differential scanning calorimetry (DSC) of all samples were performed up to a temperature of 600 °C, starting from room temperature in nitrogen atmosphere. A heating rate of 10° / min was maintained in all cases and flow rate of 30 ml / min nitrogen.

X-ray diffractometry (XRD)

Polymer samples were subjected to XRD analysis, the powdered polymer sample was packed

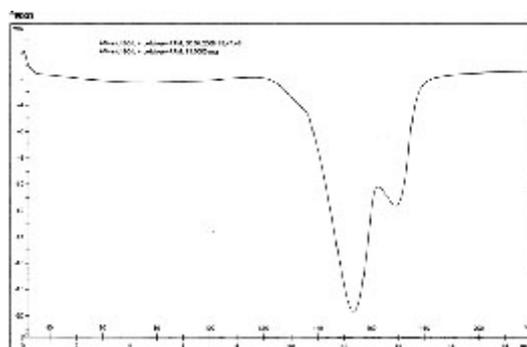


Fig. 19: DSC arid soil with (PAM+ Cellulose)

into a hole of 2 mm diameter in a small container made of perex about 1, 5 mm thick A PW 1830 diffract metre and P3020 X- Ray generator (Phillips, Holland) Were used for this study producing CuK α radiation, the scattering angle (2 θ) was varied from 10 to 45 C $^{\circ}$.

Scanning electron microscopy (SEM)

SEM micrographs were taken using Philips XL20 (Philips analytical Inc., the Netherlands). Samples was coated by gold before examination (cathode dispersion).

RESULTS AND DISCUSSION

Soils analysis

Table 2. Physical and chemical proprieties of arid soils (0-20cm)

S; Sand, C; Clay, Si; Silt, CaCO $_{3T}$; total carbonate, EC; electrical conductivity, MO; organic matter content,

CEC; cation exchange Capacity, CO; Coarse, CO.S ; Coarse Sand , CO.Si ; Coarse Silt f; fine, f.S; fine sand.

S; Sand, C; Clay, Si; Silt, CaCO $_{3T}$; total carbonate, EC; electrical conductivity, MO;organic matter content,

CEC; cation exchange Capacity, CO; Coarse, CO.S ; Coarse Sand , CO.Si ; Coarse Silt f; fine, f.S; fine sand. Clay soil had higher amount of organic mater content than sandy soil.

Elemental analysis

From 2g of Alfa plant we can obtain: Dry matter = 1.885 g, mineral matter = 1.22 %, organic matter =17.78 %.

A Viscosimetry Molecular weight of Polyacrylamide

Polyacrylamide Viscosimetry Molecular Weight, $M_v = ([\eta] / K)^{1/\alpha} = 104758, 93$ g/mol, Polymerisation degree: $DP = 104758.93/71.08 = 1473.81$

B Viscosimetry Molecular weight of Polystyrene

Polystyrene Viscosimetry Molecular

weight, $M_v = ([\eta]/K)^{1/\alpha} = 142992.2$ g/mol, Polymerisation degree: $DP = 142992.2/104.14 = 1373$.

Melting point

Melting point was measured with capillary tube using: Barnstead-Electro thermal apparatus. Polyacrylamide Melting point was: $T_f = 161$ C $^{\circ}$

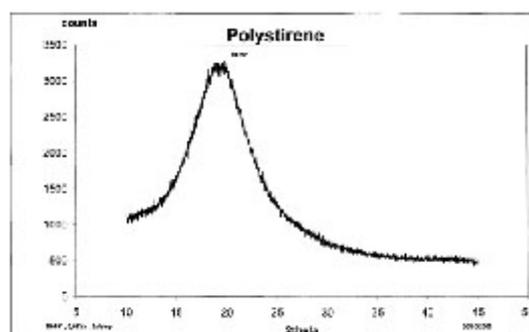


Fig. 20: X-ray diffractograms of PS

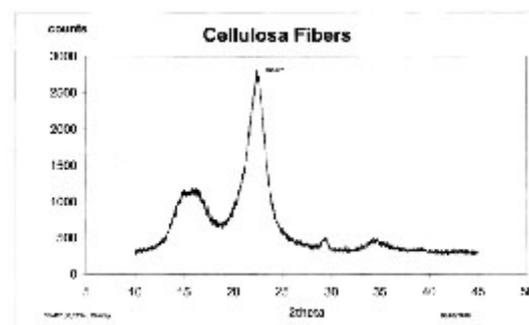


Fig. 21: X-ray diffractograms of Cellulose

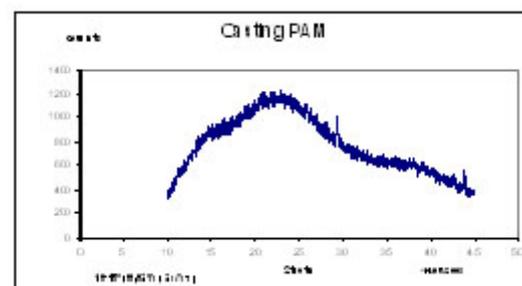
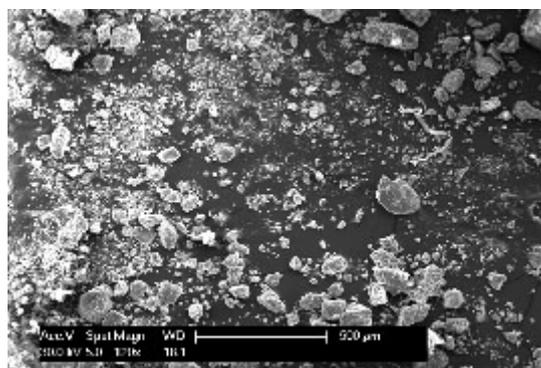


Fig. 22: X-ray

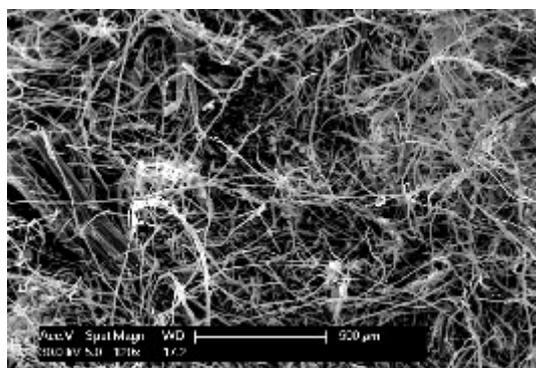
IR Spectra

From the IR spectra of Cellulose it is evident that it shows a broad absorption band at 3274 cm^{-1} (stretching frequency of the OH group), 2914 cm^{-1} (C-H stretching vibration) 1429 cm^{-1} are assigned to CH_2 vibration, 1160 cm^{-1} (cycle vibration).

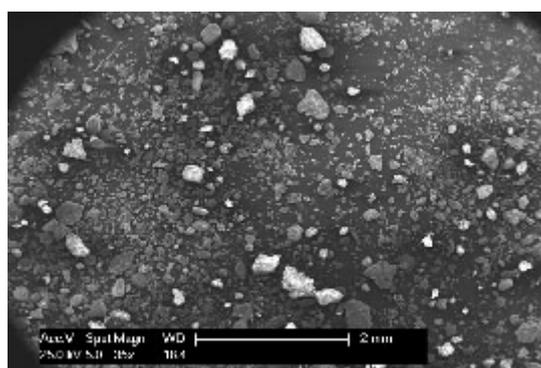
In the case of PAM a broad absorption band at 3360 cm^{-1} is for NH group, two strong bands around 1670 cm^{-1} and 1633 cm^{-1} are due to C=O and NH, 1724 cm^{-1} free acid group, 1398 cm^{-1} and 2930 cm^{-1} are for C-N and C-H vibrations, 1449 cm^{-1} (CH_2 group). In the case of Polystyrene:



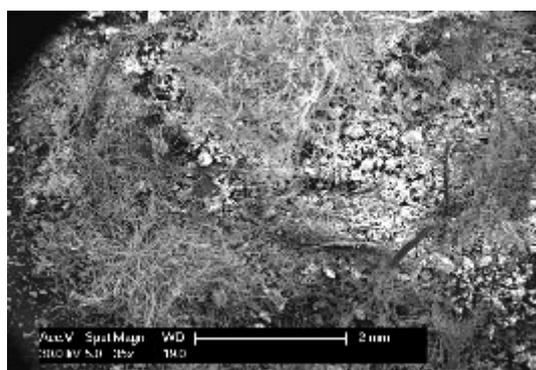
(A)



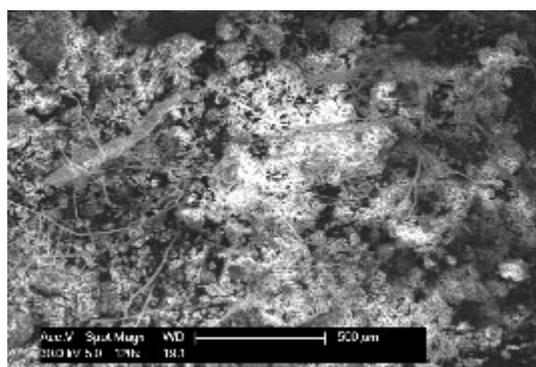
(B)



(C)



(D)



(E)

Fig. 23: Scanning electron micrographs of: (A) Semi arid soil, (B) Arid soil, (C) Cellulose Alfa-Alfa, (D) Arid soil treated with (PAM + Cellulose), (E) Semi arid soil treated with (PAM + Cellulose)

absorption band at 3028 cm⁻¹ 3061 cm⁻¹ is for (= C-H) aromatic group, 2924 cm⁻¹ vibration of C-H, 1595 cm⁻¹ vibration of (-C = C-) liaison, 1494 cm⁻¹ vibration of C-H the deformation vibration of (C-H) appear at 757 cm⁻¹ .

From the IR spectra of arid soil treated with (Polyacrylamide + Cellulose) it is evident that it shows absorption bands from 659-1000cm⁻¹ (C=C-H bending), bands from 1008.8-11842 cm⁻¹ (C-O, C-N stretching), bands from 1427.2 -1473.5 cm⁻¹ indicated the presence of (C-H bending), the bands from 1620.1 -179.6 cm⁻¹ (C=O, C=N, C=C stretching), band at 3242.1 cm⁻¹ due to OH group, band from 3487,1-3404.1 cm⁻¹ due to (N-H group), we can see OH free group at 3544.9-3616.3 cm⁻¹. From the IR spectra of semiarid soil treated with (Polyacrylamide + Cellulose): absorption band at 3622.1 cm⁻¹ Due to OH group, absorption band at 3409.9 cm⁻¹ due to N-H group. Absorption at 1421.4 cm⁻¹ indicated the presence of (C-H bending), bands from 873.7-993.3 cm⁻¹ indicated the presence of (C=C-H bending), 1062.7-1107.1 cm⁻¹ bands due to (C-O, C-N stretching).

The results of IR spectrum of arid and semi-arid soil indicated that the soils are composites consisting of Cellulose and the polymerized Polyacrylamide solution.

Thermal analysis

A-Thermo gravimetric analysis TGA

Before studying the thermal effects on these polymers, thermal stabilities and degradation patterns were determined by employing TG and DTA.

To examine the thermal stability of Polyacrylamid, Polystyrene and Cellulose Alfa-Alfa, Thermo gravimetric analysis Data under nitrogen flow were obtained: Celledulose showed initial weight loss at 70 C° is due to the presence of small amount

of moisture in the sample, the second loss is due to the loss of CO₂ and the rate of weight loss increased with increase in temperature till degradation at 350 °C.

In case of PAM: the initial weight loss is followed by a continuous weight loss with increasing temperature, the PAM decomposes in 2 stages, the polymer starts to degrade at 270 °C followed by a second stage commencing at 390 °C degradation is due to the loss of the NH₂ group in the form of ammonia .The TGA curve of Polystyrene: the first weight loss is due to the presence of small amount of moisture, then the curve still stable till 430°C temperature of Polystyrene degradation.

From figure 11: first weight loss at 50 C° probably due to humidity and the second deep weight loss at 150 C ° due to decompose of some minerals salts (calcium sulphate) then the curve is stable.

TGA curve of semiarid soil: first weight loss at 60C° due to water loss then small weight losses at 410 C° and 470 C°, the TGA decomposition

Differential Scanning calorimetry DSC

Thermal degradation was studied by differential scanning calorimetry under nitrogen flow. Cellulose shows one big endothermic curve at 80C°; from the curve the Cellulose decomposition higher then 200 °C.

In the DSC curve of PAM we can identify a glass transition in the range of 58.05-62.39 C°, a big endothermic Peak at 120°C. For Polystyrene DSC curve is more stable at high temperatures.

DSC curves of semiarid soil with (PAM + Cellulose) show a pig peak concentrated at 60-80 C° indicated the presence of Cellulose then the curve is more stable.

Table 3: Physical and chemical proprieties of Semiarid soils (0-20cm)

C	(C + Si)	CO.S	f.S	CO.Si	MO	CaCO ₃ T	EC (mS/cm)	CEC(meq /100g Soil)	PH textural	Soil
36.18	19.72	24.34	13.61	6.15	3.2	15.94	0.52	30.8	7.24	Clay

For DSC curves of arid soil with (PAM + Cellulose) we can see two endothermic peaks, the first at 155 C° and the second at 170C°.

X-ray analysis

XRD analysis showed that Cellulose in the case of PAM (Polyacrylamide) the crystalline peaks appear in the range 21- 42°.

In case of Polystyrene a crystalline peak appear at 19.65°, for Cellulose Alfa-Alfa a crystalline peak appear at 22.47° , but for casting Polyacrylamide it's amorphous.

Thickness of sub layer film

The thickness of sub layer obtained was from 0.2- 0.8 mm.

Soil water retention

In this experiment water application was controlled and no percolation losses occurred through the container, therefore any other factor that affected water accumulation was thought its effect.

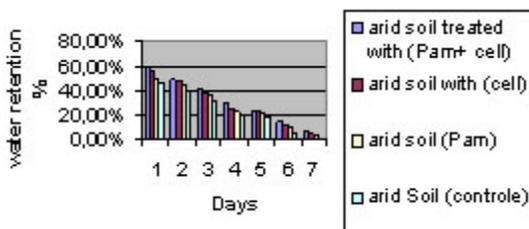


Table 3: water retention at arid soil

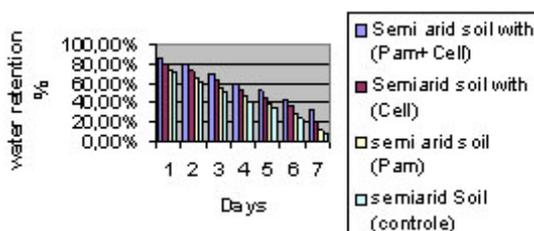


Table 4: Water retention at semi-arid soil

From Table 3 and Table 4, the amount of water retained by semi arid soil and arid soil increased by adding small amount (0.03%-1%) of polymer and biopolymer to the soil.

The water retention in semiarid soil treated with polymers and biopolymers showed better growth than in control or with only polymer or only biopolymer. NB: All the treatments had three replicates.

CONCLUSION

Different polymer and biopolymer were tested for their efficacy to retain water, augment water holding capacity, and reduce water losses by evaporation and deep percolation.

These synthetic and natural polymers show possible applicability as blend and composite on agriculture especially at desert regions to improve physical properties of soils by binding particles together and retain water.

Among prepared composites and blend polymers, the Polyacrylamide-cellulose blend showed positive results concerning water retention and improving soil proprieties.

Polyacrylamide-Cellulose mixture at arid and semi-arid soils can increase the retention of water against evaporation losses, also the synthetic Polystyrene sub layer act as a barrier to avoid water losses by deep percolation.

The water retained by polymers can be used by the plants and this addition enhanced plant growth and improved water use efficiency.

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