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## Evolution of humic fraction in a redoxi-luvisol after application of granular sewage sludge

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**INTRODUCTION.** – During the last two centuries urbanisation has led to an increasing flow of nutrients and carbon from the countryside to cities. Meanwhile, increasing cultivation of soils caused a decrease of organic carbon content, which is closely associated with soil productivity (BAUER and BLACK, 1994; ALVAREZ *et al.*, 1998). Agricultural use of biosolids is a way of returning carbon and nutrients to soils. It is also a procedure for a rational disposal of sewage sludge. Biosolids obtained from sewage sludge are source of nutrients for crops, especially nitrogen and phosphorus (PIERZYNSKI, 1994). If biosolids behave as other organic amendments, it may increase soil organic carbon and improve soil physical properties (LOGAN *et al.*, 1997).

The application of biosolids on cropping soils involves some agronomic, social and environmental problems, as well as regulation constraints (E.P.A., 1993; IAKTMENTKO *et al.*, 1995). Biosolid usually has a high water content and it is highly fermentable. Its use in rural environments frequently leads to social problems related to its unpleasant smell, and technical problems for collection and subsequent application. An alternative procedure to intent solve these problems is to subject biosolids to a process known as “heat-drying”. This process almost completely dehydrates the biosolid, and its outcome is a granular product which can be handled with the same equipment used to spread granular fertilisers.

There are few studies about the effects of the addition of sewage sludge on the amount and quality of soil organic carbon (BOYD *et al.*, 1980, HERNÁNDEZ *et al.*, 1990). Fulvic and humic acids are well known soil organic components, and they play an important role in the mobility of metallic elements within soils (STEVENSON, 1982; SPARKS, 1995). This role not only depends on the amount and proportion of fulvic and humic acids but also on their elemental composition, prevalent func-

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tional groups, and molecular weight (STEVENSON, 1982). It is not known whether "heat-drying" modifies the effects that biosolids usually have on soil organic matter content and quality.

The objectives of this study were to evaluate the effects of heat-dried biosolids on the amount of soil organic carbon and on the characteristics of humic and fulvic acid fractions of soil organic matter. It also sought to compare the effects of heat-dried biosolids and inorganic fertilisers on corn growth and yield.

**MATERIALS AND METHODS.** – A field experiment was conducted in 1997-98 at the ENSAT (École Nationale Supérieure Agronomique de Toulouse) field station, located at Poucharramet in the large alluvial corridor of the Garonne, 35 km south west of Toulouse. The soil was a redoxi-luvisol, which is a common soil in the area (GUIRESSE *et al.*, 1989). Soil main characteristics are shown in Table 1. This region has a mean annual temperature of 13.2°C, and a mean annual rainfall of 800 mm. The climate is of oceanic type subjected to both Atlantic and Mediterranean influences.

Granular biosolid used in this study was obtained from the sewage water treatment plant of the city of Toulouse. Heat-dried biosolid contained 90% dry matter. This product was obtained by heating sewage sludge at 105°C for 10 minutes. The biosolid main characteristics are shown in Table 2. Three treatments were established by the addition of granular biosolid, inorganic fertilisers, and a non-fertilised control. At the Biosolid treatment, granular biosolid was added at a rate of 11.1 Mg ha<sup>-1</sup> (dry matter basis), and immediately incorporated in the top 20 cm of soil with a cultivator. This supply of exogenous organic matter represented 28% of the organic matter naturally present in the soil. Total amounts of nutrient added were 500 kg N ha<sup>-1</sup>, 213 kg P ha<sup>-1</sup> and 36 kg K ha<sup>-1</sup>. At the fertiliser treatment, 240 kg N ha<sup>-1</sup>, 40 kg P ha<sup>-1</sup> and 71 kg K ha<sup>-1</sup> were added. Both, biosolid and fertiliser, were applied five days before sowing. Treatments were arranged in three complete blocks.

Maize was sown on May 20, 1997. Immediately after maize grain harvest (October 17, 1997) stubble was incorporated into the top soil and rye grass was sown to reduce nitrate leaching during the winter.

Nitrogen in aboveground biomass of maize was measured in Kjeldhal digest by steam distillation. Fertilisation efficiency was estimated by the difference method (DANIEL *et al.*, 1986). The difference between N uptake in control treatment and in fertiliser or biosolid treatments was considered as fertiliser or biosolid derived nitrogen (GUIRESSE *et al.*, 1999). Fertilisation efficiency was calculated as the ratio between fertiliser or biosolid derived nitrogen and total applied nitrogen. Soil samples were taken from the tilled horizon (0-0.20 m) with a drill at four dates: before the sludge was spread (April 1997), at maize harvest and before stubble incorporation (October 1997), during rye-grass growth (January 1998) and after rye-grass harvest (April 1998).

Soil samples were air dried and passed through a 2 mm sieve. The soil carbon content was determined with a dichromate-sulfuric acid mixture (NELSON and SOMMERS, 1996). Organic carbon content was transform in organic matter by using the 100/55 factor (NELSON and SOMMERS, 1996).

Total soil zinc, copper and phosphorus content were determined in aqua regia

digests by ICPMS according to ISO normalization (ISO 11466). Available P was extracted with sodium bicarbonate solution 0.5 M to evaluate available Olsen P, and determined by ascorbic acid colorimetry (Kuo, 1996).

In order to evaluate the effects of biosolid addition on soil organic matter characteristics a series of analysis were performed throughout the first year after treatment establishment. These analyses were conducted on a composite sample resulting in a mixture of three replicates of each treatment.

Soil humic substances were extracted with NaOH. Then, concentrated HCl was added to separate the precipitated humic acid from soluble fulvic acid fractions, according to BENNANI (1989) and the International Humic Substance Society (SWIFT, 1996). The humic acid fraction was redissolved by adding 0.1 M NaOH and dialysed (MWCO 1000 dialysis tub) with distilled water to purify the humic acid. The humic acid was freeze-dried. The supernatant fulvic fraction was passed through a column of Supelite™ (SUPELCO) DAX-8. The effluent was discarded and the DAX-8 column containing sorbed fulvic acid was rinsed with 0.65 column volumes of distilled water. The DAX-8 column was then eluted with 1 column volume of 0.1 M NaOH, followed by 2 to 3 column volumes of distilled H<sub>2</sub>O. The eluate was then passed through a H<sup>+</sup> saturated cation exchange resin (Amberlite IR-120 resin) and freeze dried to recover the H<sup>+</sup> saturated fulvic acid. Before freeze drying, the carbon content of both fractions, humic and fulvic acids, were determined with a TOC 5000A@Shimadzu analyser. Fulvic acid carbon was added to humic acid carbon to estimate total soil humic carbon content (HC). The procedure described above was also carried out on granular biosolid in order to extract the "humic-like" and "fulvic-like" acids. Humic substances of sludge are so called because such organic matter has undergone biotechnological process which is different from that involved in soils (GURESSÉ *et al.*, 1995a). Elemental composition (C, N and H) of the soil humic acid lyophilisates and biosolids extractions were determined by pyrolyse with a Carlo Erba 1106.

The principal chemical bonds present in the humic substances were determined by infrared analysis. Infrared spectra were obtained (with a Perkin Elmer 1600 FTIR) for the humic acid fractions from each treatment in April 1997 and in April 1998.

Other chemical properties were studied by isoelectrofocalisation (BENNANI, 1989). This method of analysis uses a pH gradient to separate families of molecules with the same pK<sub>i</sub>, on agarose gel-sorbitol (0.5g-24g/200 ml of distilled water, respectively) to which 1.2 ml Ampholine (Pharmalyte 3-10) is added. Migration characteristics were: 1500 volts, 150 milliamperes, 2 watts. Migration was over at the 1500 volt hour-stage. After the different bands of colour from black to brown have been read with a densitometer, the gel was then cut along its entire length in 5 mm-wide cubes. These cubes were placed in bottles containing 2 ml of water, shaken before the pH was measured. A range of pH was obtained so that pK<sub>i</sub> of the principal peak could be determined.

Finally, the organic molecules were separated in relation to their molecular size using Sephadex gels G100 (SWIFT, 1996) in a glass column and the various fractions of organic molecules identified by UV. The Sephadex gel G100 had a splitting range from 1000 to 100,000 Daltons. The volume of eluate recovered at the column outlet was designated VE, and the volume of the interstices into which molecules which are too large to penetrate the gel migrate, was V<sub>0</sub>. The elution of blue dextran was used to determine V<sub>0</sub> (DE HAAN, 1983). The ratio VE/V<sub>0</sub> increases as the size of the molecules (M) became smaller according to Determann's formula (DETERMANN, 1969):

$$\text{Log } M = 5.941 - 0.847 (VE/V_0).$$

RESULTS AND DISCUSSION. – The addition of both, biosolid and fertiliser, had a similar effect on maize yield. It significantly ( $p < 0.05$ ) increased from  $14.7 \text{ Mg ha}^{-1}$  in the control treatment, to  $20.9$  and  $22.6 \text{ Mg ha}^{-1}$  for the fertiliser and biosolid treatments, respectively.

Based on the different amounts of nitrogen determined in above-ground biomass at harvest for the three treatments, it was estimated that 45% of the biosolid nitrogen was taken up by the first maize crop. This value is slightly higher than those available in the literature (GUIRESSE *et al.*, 1995a; GILMOUR *et al.*, 1977). These results suggest that only approximately half of the organic nitrogen added as biosolid was available for the first maize crop. This amount was similar to the nitrogen added as fertiliser, and it could explain why both treatments had the same effect on maize yield. As both phosphorus and potassium were highly available in the soil used (Table 1), yield increase could be attributed to nitrogen supply.

Soil organic matter content was not affected by biosolid application, and remained at about 1.3% during the whole study period in all treatments. Even though carbon added as biosolid represented about 28% of the soil organic carbon, six month later all treatments shown similar levels of soil organic matter. It seems that during this period, between maize sowing and harvest, irrigation and high summer temperatures created

TABLE 1. – Some characteristics of the top soil (0-20 cm).

pH (soil: water 1:2.5)	6.0
Organic matter (%) <sup>a</sup>	1.3
Clay (%) <sup>b</sup>	15
Fine silt (%) <sup>b</sup>	24
Coarse silt (%) <sup>b</sup>	29
Fine sands (%) <sup>b</sup>	15
Coarse sands (%) <sup>b</sup>	17
Exchangeable K ( $\text{cmol}_c \text{ kg}^{-1}$ ) <sup>c</sup>	0.22
CEC ( $\text{cmol}_c \text{ kg}^{-1}$ ) <sup>d</sup>	6
Available P ( $\text{mg/kg}^{-1}$ ) <sup>e</sup>	30
Total P ( $\text{mg/kg}^{-1}$ ) <sup>f</sup>	240
Total Zn ( $\text{mg/kg}^{-1}$ ) <sup>f</sup>	28
Total Cu ( $\text{mg/kg}^{-1}$ ) <sup>f</sup>	7

<sup>a</sup> Walkley Black (NELSON and SOMMERS, 1996); <sup>b</sup> Pipette method (GEE and BAUDER, 1986); <sup>c</sup> Ammonium acetate (HELMKE and SPARKS, 1996); <sup>d</sup> Ammonium acetate (SUMNER and MILLER, 1996); <sup>e</sup> Olsen method (KUO, 1996); <sup>f</sup> Aqua regia digests, ICP-MS (JACKSON, 1958).

TABLE 2. – Composition of the granular sludge in percentage of the dry or raw (\*) matter.

	Concentration (%)
Moisture*	9.52
Organic matter <sup>a</sup>	79.88
Total N <sup>b</sup>	4.49
NH <sub>4</sub> <sup>+</sup> -N <sup>c</sup>	0.84
C/N ratio	10.7
Total P <sup>b</sup>	1.94
Total Ca <sup>b</sup>	1.78
Total Mg <sup>b</sup>	0.31
Total K <sup>b</sup>	0.32

<sup>a</sup> Titrimetric dichromate method (NELSON and SOMMERS, 1996); <sup>b</sup> Total methods (JACKSON, 1958); <sup>c</sup> Distillation method (MULVANEY, 1996).

optimal conditions for biosolid mineralisation. After maize harvest (in November 1997), maize stubble was incorporated into the soil. Stubble was 9, 11 and 13 Mg ha<sup>-1</sup> of dry matter in the control, fertilised and biosolid treatments, respectively. Despite such amounts of stubble, soil organic matter was not significantly modified at the following sampling date (January 1998). This result suggests that stubble was not yet humified. It is known that during the fall, soil temperatures in this region became less favourable for microbial activity (GUIRESSE *et al.*, 1995b).

In October 1997, the biosolid treatment showed an increment in the quantity of humic substances. This effect was not observed in later sam-

TABLE 3. – Elemental composition of the humic acid dry matter extracted from soil.

Treatment	Sampling date	FA and HA Carbon (% soil)	C %	N %	H %	C/N
Fertiliser	April 1997	0.65	34.9	3.3	4.2	12.0
	October 1997	0.39	35.1	3.4	4.6	11.9
	January 1998	0.57	36.5	3.4	4.6	12.6
Biosolid	April 1998	0.73	35.3	3.4	4.2	12
	April 1997	0.34	42.8	4.2	4.9	12.4
	October 1997	0.78	34.9	3.4	4.4	12.4
	January 1998	1.45	43.5	3.8	5.2	13.3
	April 1998	0.72	37.7	3.8	4.8	11.4

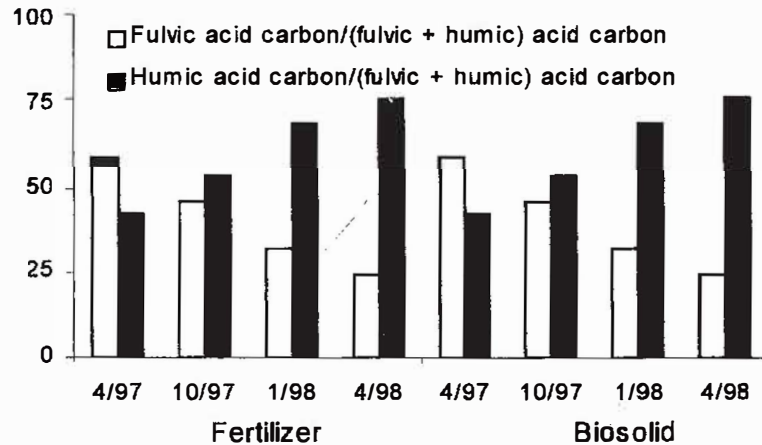


FIG. 1. – Evolution of the proportion of fulvic and humic acids in the soil during a year after biosolid application.

pling dates (Table 3). Figure 1 shows the evolution of the proportion of fulvic and humic acids in the soil during a year after biosolid application. This evolution was similar for both, fertiliser and biosolid treatments. Within this year, the proportion of humic acids steadily increased to the detriment of fulvic acids. This dynamic might be explained by the weather conditions. The two previous years before maize sowing (April 1997) were relatively wet. From October 1997 to April 1998, this wet period progressively gave way to a dry period favourable to the synthesis of humic acids (TARDY *et al.*, 1997). As they condense, small fulvic acid molecules could be precursors of humic acids (MULLER-WEGENER, 1988). This “humic” characteristic of the soil during dry periods and more “fulvic” characteristic during wet periods confirm previous observations reported by TARDY *et al.* (1997).

The elemental composition of humic substances extracted from the soil was similar at fertiliser and biosolid treatments. Nitrogen content of humic substance was between 3.4 and 4.2%, which was within the range of values observed by other researchers (BOYD *et al.*, 1980; KONONOVA, 1982; VAUGHAN, 1984), who reported values varying from 2.9 to 5.0%. Carbon content measurements in this study (34.9 to 42.8%) are slightly lower than the other Authors values (49 to 62%) (Table 3). Elemental analysis of the humic like substances extracted from the biosolid revealed a much higher nitrogen content (7.4-8.8%) (Table 4), somewhat closer to that of proteins (15-19%) (KONONOVA, 1982). However, the

TABLE 4. – Elemental composition of the humic- fulvic-like acid dry matter extracted from sludge.

	C%	N%	H%	C/N
Humic-like acid	49.1	8.8	6.9	6.5
Fulvic-like acid	48.3	7.4	6.0	7.9

addition of biosolid to the soil did not seem to have any quantitative influence in the N content or the gas C/N ratio of the humic substances of this soil six months later.

Infrared spectra were obtained for the humic acid fractions extracted in April 1997 and April 1998 from control and biosolid treatments (Fig. 2). These spectra allowed the identification of the principal functional groups of soil humic acids (Table 5). The infrared spectra showed no effect of biosolid application in the humic acid molecular structure. These spectra were very similar to those obtained by BOYD *et al.* (1980), GOMEZ and LEJEUNE (1986), GONET and ZAWALSKA (1996), HERNANDEZ *et al.* (1990) and by SCHNITZER and KHAN (1978) who showed the same main absorption bands as ours. In our study, all soil HA exhibited strong absorption around  $3420\text{ cm}^{-1}$  (hydrogen-bonded hydroxyl) and nearly  $1630\text{-}1650\text{ cm}^{-1}$  which involved all  $\text{COO}^-$ . The same amount of acidity due to  $\text{COOH}$  was shown by spectra obtained after biosolid application. The band around  $1030\text{ cm}^{-1}$  was probably due to Si-O of silicates impurities. A rather predominant absorption was visible around  $1230\text{-}1450\text{ cm}^{-1}$  involving all hydroxyl of II and III alcohols and phenols meaning that biosolid supply did not change such amount of functional groups. A moderate band about  $2930\text{ cm}^{-1}$  exhibited -CH<sub>2</sub> and -CH<sub>3</sub> asymmetric stretching which was not enhanced by sludge supply. The last band absorption from  $460\text{ to }530\text{ cm}^{-1}$  seems to imply C-H.

In the Isoelectrofocalisation analysis after migration of humic acid, a single dark brown band was observed, irrespective of the treatment. The p*K*<sub>i</sub> values was around 6.2, and no changes were produced by fertiliser or biosolid application. Clear results for the humic acid separation on Sephadex gel were only obtained with G100, which gave relatively large spectra (Fig. 3) indicating that the molecules studied were of variable but high molecular weight (between 1000 and 100,000 Daltons). At the upper limit, the use of dextran blue permitted to note the presence of molecules with weights exceeding 100,000 Daltons. At the lower limit, the molecular weights varied depending on the treatments. Two groups



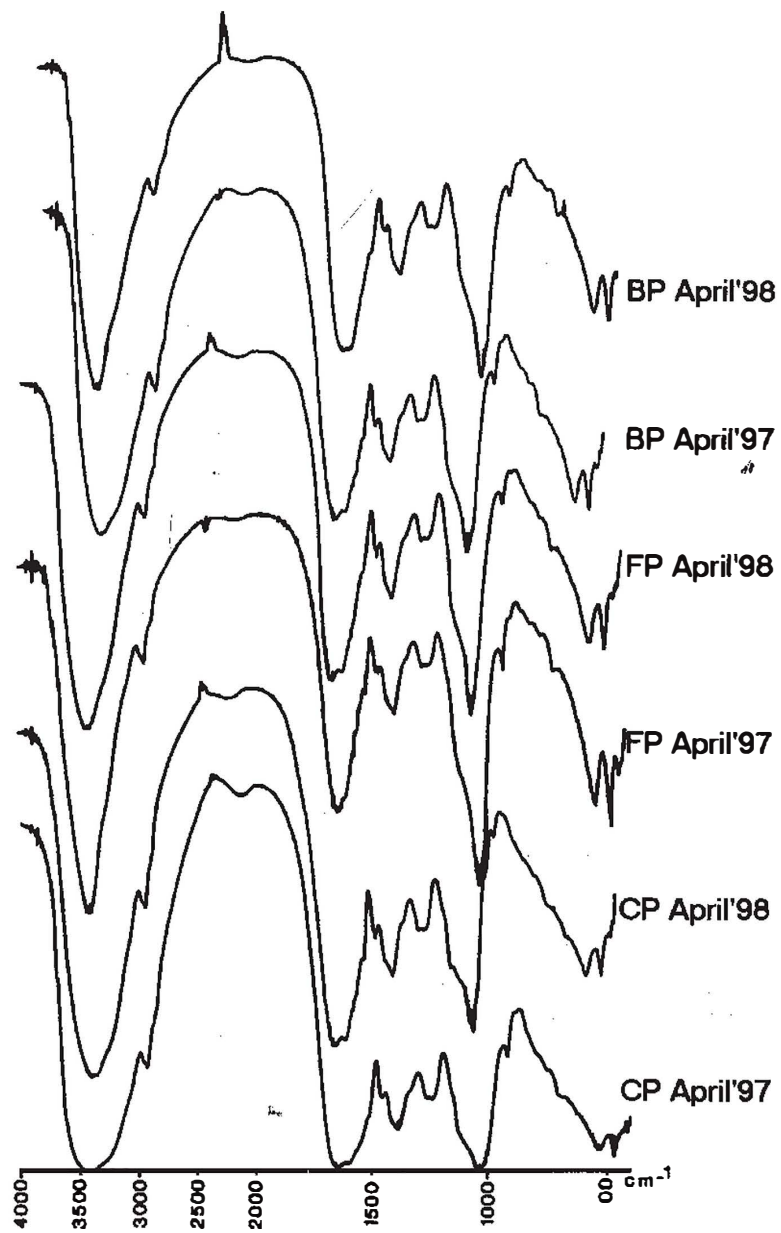


FIG. 2. – Infrared spectra of the soil humic acids in the control (CP), fertiliser (FP) and biosolid (BP) treatments at the beginning and the end of the study period.

TABLE 5. – Principal spectral bands of the humic acids from the redoxi-luvisol according to SCHNITZER and KHAN (1978).

Group	Wagging frequencies $\text{cm}^{-1}$
Hydrogen-bonded OH	3420
Aliphatic C-H stretch	2930
Aromatic C=C, hydrogen-bonded C=O of carbonyl or quinone, $\text{COO}^-$	1630-1650
Aliphatic C-H, $\text{COO}^-$ , COOH	1450
$\text{COO}^-$ , aliphatic C-H, OH from III alcohol and phenols	1400
C-O stretch of OH-deformation of COOH and OH of phenols	1233-1260
Si-O of silicates impurities (or C-O-C)	1030
Symmetric and asymmetric C-H	460-530

of molecules can be distinguished in each treated sample: a first group of very large molecules for which  $\text{VE}/\text{V0}$  equals 1 implying a molecular weight greater than 100,000 Daltons. A second family of molecules of approximately 1000 Daltons in size was also identified in both treatments. More precisely, in April 1997 the molecular sizes determined from  $\text{VE}/\text{V0}$  ratios were 2067 and 1400 Daltons for fertiliser and biosolid treatments, respectively. This molecular weight of the humic fraction from both treatments increased in April 1998 to 3700 D in fertiliser treatment and 2067 in biosolid treatment. Application of biosolid or fertiliser did not fundamentally modify the molecular weights of the humic acid fraction of these soils. However, the molecular weights of the humic acids from both treatments, when separated by gel G100 had slightly increased between April 1997 and April 1998. This result confirms that in fact greater polymerisation of humic acids occurred during this period as the climate became drier.

CONCLUSION. – Heat-dried biosolid functioned more as a source of nutrients than as a soil amendment. About half of its N content was available for the first maize crop, and it has the same effects on grain yield than an equivalent amount of N from fertiliser. Even though the amount of carbon added as biosolid represented an important part of the initial soil carbon content, biosolid application had no relevant effects neither on soil organic matter content nor on soil humic substances char-

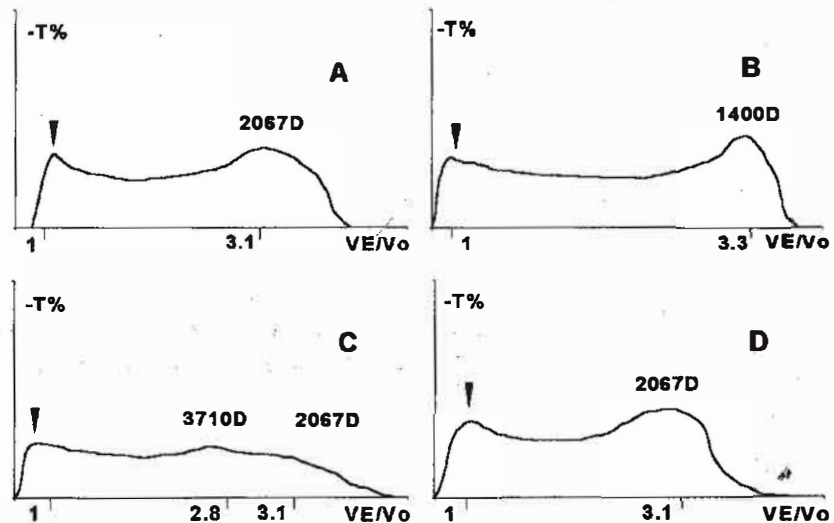


FIG. 3. - Fractionation of soil humic acid through a dextrane gel (G100), in which each molecules group appears with a lower transmission (T) and whose molecular size decreases when the volume of eluate at the column outlet (VE) and the volume of the interstices of the gel (V0) ratio (VE/V0) increases. A: Fertiliser treatment in April '97; B: Biosolid treatment in April '97; C: Fertiliser treatment in April '98; D: Biosolid treatment in April '98.

acteristics. Therefore, at the rate used in this experiment, heat-dried biosolids may be treated as a nutrient source for crops, and no effect on soil organic matter quantity and quality could be expected from its use. The fact that heat-dried biosolids had the same effects on maize yield than mineral fertilisers, and no effects on soil organic matter supported the conclusion that heat-dried biosolids should be used as a fertiliser and not as a soil amendment.

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**SUMMARY.** – Agricultural use of biosolids is a way of returning carbon and nutrients to soils, and a rational disposal of sewage sludge. Its use in rural environments faces some problems related to its high water content and readily fermentable status. An alternative procedure is to heat-dry the biosolids, which results in a granular product that can be handled with the same equipment used to spread granular fertilisers. A field experiment was carried out on a redoxi-luvisol in Toulouse (France) to evaluate the effects of the application of heat-dried biosolids on the amount of soil organic carbon and on the characteristics of humic and fulvic acid fractions of soil organic matter. It also sought to compare the effects of heat-dried biosolids and inorganic fertilisers on corn growth and yield. Three treatments were established by the addition of granular biosolid, inorganic fertilisers, and a non-fertilised control. Maize was cropped, and soil organic matter content and some humic substances characteristics were monitored during a year after biosolid application. Even though the amount of carbon added as biosolid represented 28% of the initial soil carbon content, biosolid application had no relevant effects neither on soil organic matter content nor on soil humic substances characteristics. Humic acid elemental composition, molecular size, and structure were not affected. Biosolid carbon seems to have readily mineralised during the crop cycle, so it functioned as a nutrient source without affecting any soil organic matter quality.