Changes in the Mineralisation of Nutrients and Sunflower Biomass in Soil Irrigated with Water from Oil Exploration in a Semi-Arid Environment

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Abstract—Wastewater from oil fields may be an option for irrigation, especially in regions which have low rainfall with high variability. The aim was to evaluate the composition and decomposition of shoot biomass from sunflower plants irrigated with water from oil wells, which had been subjected to filtering (FPW) and reverse osmosis (OPW), using groundwater (UGW) as a reference. Two tests were then carried out to evaluate decomposition of the residue. In the first test, residues produced with FPW, OPW and UGWwere incubated in soil and irrigated with groundwater (UGW). In the second test, residues from plants irrigated with UGW were also incubated, but irrigated with FPW, OPW and UGW. Significant differences were seen in the levels of Na, Mg and lignin in the residues with the use of FPW, showing greater levels for Na, and lower levels for Mg and lignin. The loss in biomass of the incubated residues was not significant in either test; this was not seen in the Mg e Nwith smaller losses than the biomass, or theNa, K e S with greater losses, especially when produced with FPW and OPW respectively. In the residue produced with UGW, differences were identified for Ca and Na, with the order of losses for type of water being UGW=FPW> OPW and OPW=UGW>FPW respectively. Irrigation using water from oil extraction alters the chemical characteristics of the soil and the composition of cultivated plants at a level sufficient to influence the rate of decomposition of the organic residue.

Keywords— Sunflower residue, Produced water, Wastewater management, Mineralisation, Substrate quality.

I. INTRODUCTION

The use of wastewater has become an acceptable agronomic practice (Caovillaet al.2010; Sandriet al., 2006), being of considerable interest to the oil industry as it removes the problem of disposing of produced water, helps to conserve water resources, and improves nutrient recycling (Chatzakiset al. 2011; Medeiroset al. 2008). However, the produced water which is generated in oil wells,may contain heavy metals, organic and inorganic compounds (Al-Haleemet al. 2010), salts, and additives used during extraction, such as anticorrosives and biocides (Nascimentoet al. 2006), which can pose risks to the environment.

A large volume of produced water is generated in an oil field, and may be an option in the irrigation of crops grown for fuel. Irrigation with produced water can be particularly effective in areas with poor rainfall distribution and a shortage of drinking water. However, this can alter the chemical properties of the soil (Chatzakiset al.2011; Guptaet al.2010; Lado andBen-Hur 2009; Sharmaet al, 2007) and as a consequence, the chemical composition of plants (Bhattacharyaet al.2010; Guptaet al.2010; Il'In 2007; Sharmaet al.2007).

The decomposition of organic matter is an important stage in nutrient cycles, and is affected by the chemical composition of the plant residue, as nutrient levels and the energy available to decomposers determine the efficiency of the mineralisation of organic residues (Manzoniet al. 2010; Minderman 1968). Studies into decomposition have shown a reduction in the mineralisation of organic residue for levels of Mg, while the opposite was seen for Ca (Lemmaet al. 2007) and P (Birousteet al.2011; Goebelet al.2011). Talbot and Treseder (2012) reported that initial levels of N in organic residue increased mineralisation, while Birousteet al. (2011) were unable to confirm this observation. On the other hand, initial concentrations of lignin were seen to negatively influence losses in biomass (Amougouet al.2011; Talbot and Treseder 2012) but had no effect on the release of N (Talbot and Treseder 2012).

Changes in soil properties, in particular increases in the concentration of toxic minerals, can also affect the decomposition of organic residue, since they affect the structure and activity of microbial communities in the soil (Aceveset al. 1999; Chander and Bookes 1991). The physical and chemical properties of the soil are known to influence microbial communities (Bowleset al. 2014; Rietz and Haynes 2003). This effect can be modified by salinity, sodicity and alkalinity, which can reduce biomass and microbial activity (Yuanet al. 2007), and inhibit respiration (Maviet al. 2012).

It is probable that irrigation with produced water from oil extraction causes changes in the chemical properties of the soil and the chemical composition of plants. Information is available on the effects of chemical composition, both of the soil and of organic residue, on rates of decomposition (Amougouet al.2011; Talbot andTreseder2012; Uselmanet al. 2012.), but no study has evaluated the effects of water quality on the chemical composition of plants, and the consequent changes in the mineralisation of nutrients and the decomposition of biomass, cellulose and lignin. Crop residues are important for the nutrient-cycling process in systems of agricultural production; it is therefore essential to evaluate any possible changes in the decomposition of plants irrigated with produced water. In the present study, decomposition rates for the residues of sunflower shoots (Helianthus annuus L. cv. BRS 321), irrigated with produced water subjected to filtration and reverse osmosis, and with groundwater captured in the Açu aquifer, were studied. The aim was to determine whether produced water submitted to two different treatments (filtering and reverse osmosis) alters the chemical characteristics of plants and influences decomposition of organic residue.

II. MATERIALS AND METHODS

The study area was an experimental field of the Brazilian oil company, Petrobras, located on the Belém Farm, in Aracati, in the State of Ceará, in the semi-arid region of Brazil, (4°43'6" S, 37°32'48" W). Average annual temperature and rainfall in the region are 28°C and 949.2 mm respectively, with the greatest concentration from March to May. Profiles were described for the study area, and the class of soil identified as aHaplic Arenosol (FAO 2006).

Two crop cycles of the sunflower *Helianthus annuus* L., cv. BRS 321 were conducted in an experimental design of

randomised blocks, with three replications in plots of 400m². In the first growing period, the crop cycleran from July to Octoberof 2012, and in the second, from March to June of 2013. The plots were irrigated with wastewater from oil production, which were subjected to two pre-treatments after extraction of the oil. For the first pre-treatment, the water was initially filtered through sand filters, and then passed through a cation-resin filter to remove residue of the caustic soda used in the oil-water separation process (FPW). In the second pre-treatment, the FPW was subjected to nanofiltration and reverse osmosis (OPW). The control treatment used groundwater captured from wells at a depth of 250 m in the Açu aquifer (UGW). The chemical characteristics of the irrigation water are shown in Table 1.

A drip irrigation system was used, with the emitters distributed along the crop rows, at a spacing of 0.30 m and with a flow of 1 L h⁻¹. In order to meet the water requirement of the crop, the amount of water applied to the soil was up to 4.5 L m⁻² day⁻¹, calculated based on the evapotranspiration of the sunflower crop and water loss through drainage, employing columns of mini-lysimeters in the experimental plots. During the first growing period, on average 271 L m⁻²OPW, 365 L m⁻²FPW, and 393 L m⁻ ²UGW were applied for plant irrigation. During these cond period, 395 L m⁻² OPW, 353 L m⁻² FPW, and 260 L m⁻² UGW were applied.During the experiment, the maximum mean temperature was 33°C, with a minimum f 23°C, and a precipitation of 483 mm (L m⁻²)in the second growing period.Based on the soil analysis prior to planting, it was necessary to correct the soil to meet the nutritional requirements of the crop. The soil was thereforecorrected with organic fertiliser, 7.5 kg/lm before the first crop, and 2.5 kg/lm before the second crop. Also, in each cycle, doses of 80 kg/ha P2O5 and 40 kg/ha K2O were incorporated into the soil before planting, as well as 50 kg/ha N close to the flowering stage.

Samples of shoot residue from sunflower plants produced in the first crop cycle, and irrigated with OPW, FPW, and UGW, were incubated with UGW in the irrigated plots (Test 1); and plant residue from plots where only UGW was used was incubated in the plots irrigated with OPW, FPW and UGW (Test 2).

For incubation, 30 g of air-dried shoot residue with a maximum size of 0.05 m, were placed into 0.14 m by 0.15 m nylon bags of anti-aphid mesh (Thomas and Asakawa 1993) and arranged horizontally in the soil at a depth of 0.05 to 0.10 m, near the drippers, avoiding direct contact with the plant roots. The chemical characteristics of the 0 to 0.1 m layer of soil (which corresponds to the depth of the incubated residue) after irrigation with the different types of water, are shown in Table 1. The bags were collected after 14, 28, 41, 55 and 69 days of incubation,

and the residue dried at 65°C, weighed to determine the biomass, and stored for further chemical analysis. Subsamples of the crop residue were used to determine the dry weight (65°C) and chemical characteristics at the start of the experiment (t = 0). For each trial, 45 nylon bags containing residue were incubated in the soil, considering three treatments (OPW, OPF and UGW), five collection periods (14, 28, 41, 55 and 69 days of incubation) and three replications (n = 3).

The collected residue was submitted to nitro-perchloric digestion (3:1 v/v) and the levels of Ca and Mg determined by atomic absorption spectrophotometry (Analyst 400, PerkinElmer), the Na and K content was determined by flame photometry (DM-62, Digimed), and S and P determined using spectrophotometry (Femto 600 Plus). Levels of N-NH₄ were determined by Kjeldahl distillation (Silva 2009), and TOC was quantified by wet digestion with potassium dichromate and H₂SO₄ while heating (Yeomans andBremner, 1988). Lignin and cellulose levels were also determined, using the method of acid detergent fibre (ADF) (Goering and VanSoest, 1975). All the levels were calculated by multiplying the above concentrations by the weight of the collected residue.

At the end of the 69 day incubation period, losses were estimated for the biomass, minerals, lignin and cellulose for each situation under study, the half-life (t) for each of these being determined with the equation t = ln(2)/k (Rezendeet al. 1999), where k is the decay constant obtained from the equation Xt-X0.e^{KT} (Thomas and Asakawa, 1993).

The data were subjected to the Shapiro-Wilk test for normality, and Bartlett's test for the homogeneity of variances, to verify that the assumptions of the variance analyses were met. After noting the normal distribution of the variables, analysis of variance (ANOVA) was used to determine the statistical differences (P<0.05) between the mean values for the loss of biomass, nutrients (Ca, Mg, Na, K, S, P, and N), C, cellulose and lignin at the end of the 69 day incubation period. Mean values for data displaying any variation were compared by Tukey's test at a level of 5%.

To identify the relationship between the chemical characteristics of the residue and the loss of biomass, nutrients, lignin and cellulose, multivariate analysis of variance (MANOVA) was performed. To identify which variables (chemical characteristics of the residue) were more important in controlling decomposition, the value for Wilks's lambda was calculated; this allows evaluation, for each variable, of the statistical differences for the mean values between groups. The value for Wilks's lambda varies between 0 and 1; the smaller this value, the greater the discriminatory power between sets

of variables. Due to interference from the chemical composition of the residue, and the combination and proportions of the different constituents of the decomposing material, the predictor variables used in the model were the initial values for Ca, N, P, K, Na, S, Mg, C, lignin and cellulose, and the ratios of C:N, cellulose:Ca. cellulose:N. cellulose:Pcellulose:K, cellulose:Na, cellulose:S, cellulose:Mg, lignin:Ca, lignin:N, lignin:P, lignin:K, lignin:Na and lignin:Mg. The response variables were the weight-loss percentage (%) for Ca, Mg, Na, N, P, K, S, C, cellulose, lignin and biomass in organic residue seen at the end of the incubation period (69 days). Statistical analysis was carried out using the R software (R Core Team, 2013).

III. RESULTS

Chemical composition of the plant residue

The nutrient concentrations and compounds from the biomass of the sunflower shoots exhibited distinct behaviours when irrigated with the different types of water. Significant differences (P<0.05) were found in the levels of Na, Mg and lignin, with different behaviour when using FPW, the highest levels being seen for Na, while Mg and lignin displayed the lowest values. S and P were significantly lower, while lignin levels were higher compared to UGW when OPW was used. For the other elements and compounds (Ca, K, N, C and cellulose) there was no effect from the different types of water used, and no significant statistical differences were noted (P<0.05) (Table 2).

Decomposition of shoot residue fromsunflowers irrigated with different types of water (OPW, FPW and UGW),and incubated insoil irrigated with UGW (Test 1)

The loss percentage for biomass in the residues produced with OPW, FPW and UGW was around 73%, with no significant difference (P<0.05) when irrigated with UGW (Table 3). The same behaviour was also seen for P, Ca, C, cellulose and lignin, with overall mean values of 52, 50, 80, 80 and 44% respectively. However, this lack of significance in the differences was not found when evaluating the remaining nutrients, identifying loss percentages which were smaller (Mg, P and N) and greater (Na, K and S) than the mean for biomass (the main reference, due to being composed of these nutrients), and highlighting the significant statistical differences for the losses of Na, Mg and K. The losses were higher for Na and Mg when the residues were produced with FPW and OPW respectively. K had the greatest losses among all the elements studied, on average 98%, with similar losses whether the residue was irrigated with FPW or UGW, these losses being greater than for

OPW. S and N also showed significant losses, but in the following order for the type of water used for irrigation: UGW>OPW=FPW and FPW>OPW=UGW respectively. It was found that generally the greatest losses correspond to the smallest values for half-life. Again,this assertion can be made considering the biomass as reference, as was done with the loss of nutrients. Elements withlosses greater than the average seen for biomass, had the lowest values for half-life, while those with smaller losses, had the longest half-life.

There are statistical differences between the values for half-life of the residues when irrigated with the different types of water (Table 3); here, the use of OPW is highlighted, since the half-lifewas longer using that type of water, as was the case with Na, K, S, N and the biomass. Some cases showed similar results when using the other types of water (UGW and FPW), however no general trend was seen. In the case of Mg, the use of FPW and UGW gave the greatest value for half-life, while the half-life of P was greater in residue produced with UGW.

Decomposition of shoot residue from sunflowers in the area of UGW, and incubated in soil irrigated with different types of produced water (OPW, FPW and UGW) (Test 2)

The loss of biomass in this test (73%) was similar to the previous test (72%) with no statistical differences for type of water used for irrigation in the residue produced with UGW (Table 4). The same trend was seen for some nutrients and compounds of the biomass, for example Mg, S, P, K, C and N, cellulose and lignin. The nutrients which exhibited statistical difference were Ca and Na, however no similarity was seen between their behaviour, with the order of losses for type of water used for irrigation being: UGW=FPW>OPW and OPW=UGW>FPW respectively.

Half-life was more sensitive in indicating variations in decomposition, as there were significant statistical differences for Ca, Mg, Na and biomass (Table 4). There was statistical similarity between FPW and UGW for half-life in Ca and Mg, however FPW gave a greater half-life for Na with a shorter half-life for biomass. OPW and UGW showed similarity between Na and biomass for half-life, with OPW giving a shorter half-life for Mg and a longer half-life for Ca.

Influence of the chemical composition of the residue on decomposition and the loss of biomass and nutrients

The loss of nutrients and organic compounds was influenced by the chemical properties of the residue under decomposition (MANOVA, F=12.85, R²=0.85, P<0.001). Considering the effect of the predictor variables group

(residue composition) on the variable response group, the C:N ratio displayed the greatest control over the loss of nutrients and organic components, as indicated by the lower lambda value (Table 5), followed by the cellulose:Mg ratio. The C:N ratio influenced the loss of Mg, Na, N, S, biomass, lignin and cellulose, while the cellulose:Mg ratio affected the loss of Mg, Na, biomass and cellulose. The loss of Na and cellulose, and of N and S, were also influenced by the ratios of cellulose:N and cellulose:S, respectively.

IV. DISCUSSION

The composition of the treated water (FPW or OPW), may be related to changes in the levels of the same elements when also evaluated in the soil, i.e. reductions or increases in the levels of these elements in the water correspond to similar behaviour in the soil (Table 1). The change in soil properties (salinity, for example) may be associated with changes nutrients found in sunflowers tissues.Despite these variations in the soil being relatively wide for the type of water/treatment, only the levels of Na, Mg and lignin showed significant changes in the tissue. Variations in the levels of Na in the different types of water used for irrigation may be associated with the different levels of Na (Shahbazet al. 2011), Mg (Liu et al. 2010) and lignin (Wanget al. 1997) found in the sunflower tissue. These findings also underline the efficiency of the adopted treatments, as regards the presence of elements and the effect on the soil and plants, with a clear advantage seen withOPW, where their composition displays a reduction in levels. Similar results for the influence of the type of water on the soil, and the efficiency of the wastewater treatments, were discovered by Morugán-Coronado et al. (2011).

However, the rates for loss of mass and half-life in the residue, when compared to the overall average biomass in both tests under study, were very similar, which demonstrates that if evaluated using only biomass, control of residue decomposition should be attributed to the environment and its conditions (humidity, wind, sunlight, microbial activity, etc.). This association will be real; but it is also necessary to consider variations in the composition of plant tissue and the type of water that was used in producing the residue, since, in the two situations under study (Tests 1 and 2), there was an effect on the loss (%) and half-lives (t) of the nutrients, and on the differences in the loss of total biomass for the residue in decomposition. These results are consistent with results obtained in studies carried out by Amougouet al. (2011), Lemma et al. (2007), and Uselmanet al. (2012), in which those authors observed that the chemical composition of the residue resulted in variations in its mineralisation.

When considering the reference conditions (UGW), changes in the residue produced with FPW were enough to increase the loss of Na and K, whereas the changes that occurred in the residue from plots irrigated with OPW were enough to increase the loss of Mg and P, and reduce loss of S. Compared to the residue produced with OPW, the loss of Na, K, S, N, and biomass was greater in residue obtained with FPW. It can therefore be demonstrated that irrigating with produced water alters the chemical composition of the sunflower, and subsequently influences mineralisation of the plant residue.

Previous studies have demonstrated the isolated effects of the initial chemical composition of plant residue on the decomposition of such components of organic residue as C and lignin (Amougouet al. 2011; Talbot and Treseder 2012), and Mg, P, and Na (Goebelet al.2011; Ranjbar andJalali 2012), or on the ratios of C:N, lignin:N and lignin:P (Osono and Takeda 2004; Silver and Miya 2001). In the present study, the loss of total biomass, Na, Mg, S, N, C, cellulose and lignin was mainly influenced by the C:N ratio. However, the ratios of cellulose:Mg, cellulose:N and cellulose:S also influenced decomposition of the sunflower residue (Table 5), possibly due to the decomposer organisms in the soil used the cellulose as a C source (Castroet al. 2010). For decomposition of structural components such as cellulose, high levels of nutrients are required (Talbot andTreseder 2012), which may explain the results found in this study, since the ratio of cellulose to nutrients affected the rate of decomposition, which did not occur when cellulose was considered in isolation.

As demonstrated by the MANOVA analysis,the cellulose:Mg ratio was the most important in controlling the rate of decomposition of the sunflower residue than the ratios of cellulose:S and cellulose:N, as it showed the lowest value for lambda ($\lambda = 0.053$, P<0.05; Table 5). It is possible that the chemical fertilisation of the soil during preparation of the area met the needs for N, P and K of the microorganisms in the soil, thus not depending on the nutrient content of the residue during decomposition of the organic matter. However, this relationship is not yet clear; new studies could therefore consider residues with different cellulose to nutrient ratios, in evaluating the rate of decomposition.

In contrast to results obtained in otherstudies (Amougouet al. 2011; Talbot and Treseder 2012) there was no effect from lignin content on the loss of nutrients, C, cellulose or total biomass. It is possible that the incubation period of the residue (69 days) was not sufficient for the lignin to affect decomposition, or it can be considered that interference by the lignin in the rate of decomposition only occurs after depletion of the more labile fractions of the organic residue, as suggested by Silver and Miya (2001).

There were similar variations in the loss of some residue components in soils irrigated with UGW and with produced water (OPW and FPW). However, irrigating the soil with OPW or FPW favoured the loss of Ca and Mg respectively. High levels of salts affect the decomposition of organic residue by reducing the size and diversity of the microbial community in the soil (Chowdhuryet al.2011; Rietz and Haynes 2003; Yan andMarschner 2013; Yuanet al., 2007) as well as its activity (Hagemann 2011; Maviet al. 2012). But in this study, the highest values seen for Na⁺, Cl⁻, HCO₃₋ and EC with FPW (Table 1) did not reduce the loss of nutrients (except for Na), biomass or other constituents of the sunflower residue. Under these conditions, Bowles et al. (2014) and Yan and Marschner (2013) associate the capacity of soil microorganisms for rapid response with changes in soil salinity;this can be attributed to adaptation to the new conditions. Also to be considered are the joint changes in the microbial structure of the soil due to salinity and alkalinity, as observed by Yuan et al. (2007) in arid soils, resulting in selection of the most efficient species for promoting decomposition of the residue. In order to clarify this issue, further studies are needed into microbial communities involved in the decomposition of residue in soils irrigated with FPW.

V. CONCLUSIONS

Irrigation with produced water changes the chemical characteristics of the soil and the composition of cultivated plants at a sufficient level to influence the rate of decomposition of the organic residue, these effects being variable and dependent on the type of pre-treatment used. The produced water treated by filtration favoured greater adecomposition of sunflower residue than that by reverse osmosis.

It is necessary to test new ways of treating produced water to be used in the irrigation of crops, especially processes where there is no addition of biocides, in the case of treatment by reverse osmosis, and which are effective in the removal of salts, in the case of treatment by filtration. Studies are also necessary to evaluate the cumulative effect of successive irrigation with produced water on the decomposition of residue and on the soil microbiota, as well as on the accumulation of toxic minerals in the soil.

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