ORGANIC MATTER FRACTIONS AND QUALITY OF THE SURFACE LAYER OF A CONSTRUCTED AND VEGETATED SOIL AFTER COAL MINING. II - PHYSICAL COMPARTMENTS AND CARBON MANAGEMENT INDEX

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ABSTRACT

Soils constructed after mining often have low carbon (C) stocks and low quality of organic matter (OM). Cover crops are decisive for the recovery process of these stocks, improving the quality of constructed soils. Therefore, the goal of this study was to evaluate the effect of cover crops on total organic C (TOC) stocks, C distribution in physical fractions of OM and the C management index (CMI) of a soil constructed after coal mining. The experiment was initiated in 2003 with six treatments: Hemarthria altissima (T1), Paspalum notatum (T2), Cynodon dactylon (T3), Urochloa brizantha (T4), bare constructed soil (T5), and natural soil (T6). Soil samples were collected in 2009 from the 0.00-0.03 m layer, and the TOC and C stocks in the physical particle size fractions (carbon in the coarse fraction - CCF, and mineral-associated carbon - MAC) and density fractions (free light fraction - FLF; occluded light fraction - OLF, and heavy fraction - HF) of OM were determined. The CMI components: carbon pool index (CPI), lability (L) and lability index (LI) were estimated by both fractionation methods. No
INTRODUCTION

Coal is a major source for power generation in the world. In Brazil, the main coal reserves are found in the States of Rio Grande do Sul (RS) and Santa Catarina (SC), with 28.8 and 3.36 billion ton, respectively. In RS, approximately half of the State’s reserves (12.28 billion ton) lie in Candiota alone, making it an important center of power generation. In this municipality, coal is exploited in open pits, by the mining company Companhia Riograndense de Mineração (CRM) (Santucci, 2009).

After mining, the soils are “constructed” from mine tailings and the surface horizons previously removed from the original soil. Among the various soil chemical, physical and biological properties affected negatively by the mining process, the loss of soil organic matter (OM) is the most influential, aggravated by erosion and reduction of soil aggregation and physical protection of OM. In addition, the dilution of OM levels as a result of the mixing of the horizons (A, B, C) during soil construction, commonly results in OM levels below 1% in the surface layer of constructed soils (Corrêa, 2009). Organic matter is a key indicator of changes in soil quality. Frequently, these changes were detected with greater sensitivity by physical and/or chemical OM fractions than by total OM (Santos et al., 2012). Thus, the study of these fractions can indicate in advance the best-suited treatment for the recovery of constructed soils.

Keywords: recovery of degraded areas, cover crops, carbon stocks, lability.
Areas adjacent to the study, undisturbed by human action or under degradation, are normally used as control (CMI = 100).

The CMI was originally proposed by Blair et al. (1995), who obtained labile OM fractions by KMnO₄ oxidation. Due to the limitation of this technique and the greater effectiveness and simplicity in determining OM fractions by physical fractionation, some authors proposed the use of physical fractions as a mean to estimate CMI of soils under different agricultural cropping systems. In this sense, Dieckow (2003) adapted the method proposed by Blair et al. (1995) and estimated CMI from OM physical fractions. The authors found a higher CMI and therefore higher soil quality in cropping systems with leguminous species than in systems with grasses only. Vieira et al. (2007) observed a greater sensitivity of physical particle-size and density fractionation (NaI, 1.8 kg dm⁻³) in determining the labile OM fraction than of the conventional chemical method proposed by Blair et al. (1995), resulting in a more accurate CMI estimate in agricultural fields under no-tillage (NT) and different cropping systems.

Recently, Conceição et al. (2014) compared the methods of particle-size and density fractionation (NaI, 1.8 kg dm⁻³ and sodium polytungstate - PTS, 2.0 kg dm⁻³) with regard to the efficiency in determining OM lability and estimating CMI in soil under conservation tillage. The authors observed greater OM lability and CMI in soil under NT and crop rotation with grasses and legume species than in soil under conventional tillage (CT) and without legumes. The higher amount of residue and enhanced soil protection in the NT system increased the quantity and lability of OM. Independent of the solution (PTS or NaI) used in the density fractionation to obtain the labile OM fraction and estimate CMI, this index was highly correlated with the CMI estimate based on OM particle-size fractions. However, since the labile OM fraction extracted by the PTS solution was higher, this was more appropriate than NaI solution for density fractionation.

Physical fractionation has been frequently used in the study of OM of soils under agricultural cultivation systems. However, even in agricultural soils, few studies use density fractionation as a basis for estimating CMI, but rather particle-size fractionation. In constructed soils, studies focused on the behavior of particle-size and density fractions of OM are even rarer. In these soils, CMI could be a key indicator of measures to increase the OM quality and quantity, accelerating the soil restoration process. Therefore, the purpose of this study was to evaluate the effect of different perennial grasses on C stocks in physical particle-size and density OM fractions of a soil constructed after coal mining, and to estimate the CMI from OM fractions obtained by both methods.

### MATERIAL AND METHODS

#### Description of the experimental area and soil sampling

The experimental area is located in a mining area of CRM in Candiota, RS. The regional climate is classified as Cfa (humid subtropical), according to Wilhelm Köppen, and the mean annual temperature and rainfall are 17 °C and 1,400 mm, respectively. The soil in the field is characterized by a deeper layer consisting of fragmented material of mudstones, shales, carbonaceous shales, and of sandstone, removed by blasting the upper coal layer. The surface layer with variable thickness (30-40 cm) consisted predominantly of the B horizon soil removed prior to coal extraction and had a clayey texture (Table 1).

The natural soil (unmined) used for comparison, was classified as Alfisol (NRCS, 2010), with a loamy texture in horizon A (Table 1). For chemical properties of the experimental soil, see Santos et al. (2008).

The experiment was established in November/December 2003, in a randomized complete block design, with four replications (20 m² plots) and four grass species, in the following treatments:

| Treatment   | Clay   | Sand  | Silt   | Textural class | Bd      |
|-------------|--------|-------|--------|----------------|---------|
|             | g kg⁻¹ |       | g kg⁻¹ |                | kg dm⁻³ |
| T1 - Hemarthria | 463.7  | 298.1 | 238.3  | Clay           | 1.36    |
| T2 - Paspalum   | 474.2  | 291.9 | 233.9  | Clay           | 1.46    |
| T3 - Cynodon    | 469.7  | 283.8 | 246.8  | Clay           | 1.43    |
| T4 - Brizantha  | 452.6  | 289.7 | 257.7  | Clay           | 1.41    |
| T5 - Bare soil  | 456.7  | 311.7 | 231.5  | Clay           | 1.46    |
| T6 - Natural soil| 227.2  | 483.6 | 289.1  | Loam           | 1.47    |
[Hemarthria altissima (Poir.) Stapf and C. E. Hubbard] (T1); Paspalum notatum Flügge (T2); [Cynodon dactylon (L.) Pers.] (T3); and [Urochloa brizantha (Hochst.) Stapf] (T4); T1 and T3 were planted from seedlings and T2 and T4 from seeds, as recommended for each species.

When the experiment was installed, the soil was chiseled to 0.10-0.15 m, 10.4 Mg ha⁻¹ of lime was applied (relative power of total neutralization 100 %) and the soil fertilized with 900 kg ha⁻¹ of the formula 5-20-20 (NPK) as recommended, according to Cambardella and Elliot (1992). Sodium hexametaphosphate (60 g L⁻¹) was added to 20 g of air-dried soil. The suspension was shaken for 15 h in a horizontal shaker (150 oscillations min⁻¹). Then the suspension was washed with water on a sieve (mesh size 0.053 mm). The C of the retained material represents the C in the coarse fraction (CCF), while the C contained in the material that passed through the sieve corresponds to the C associated to minerals (MAC). The total organic carbon (TOC) and CCF contents were determined by dry combustion (TruSpec CHN Elemental Analyzer), whereas the MAC content was calculated as the difference between TOC and CCF.

The TOC and C stocks in the fractions CCF and MAC were computed from the C contents in each fraction and Bd. From these data, the distribution of CCF and MAC was calculated as percentage of the TOC stock.

### Physical density fractionation of OM

The physical density fractionation of OM was performed according to Conceição et al. (2008). To 10 g of soil (in weighed proportions of aggregates <2.00 mm and aggregates 9.52-2.00 mm), 80 mL of PTS solution (2.00 kg dm⁻³) was added. The centrifuge tube containing the suspension was shaken gently by hand and then the suspension was centrifuged for 1 h (2000 g). After centrifugation, the supernatant containing the free light fraction (FLF) was vacuum-filtered through previously weighed glass-fiber filters. The PTS solution was reinserted in the tube together with the remaining material and the suspension was dispersed by sonication. The adequate energy for maximum aggregate dispersion was previously determined for the three situations of the experiment: constructed soil with cover crops (T1, T2, T3, T4) - 430 J mL⁻¹, bare constructed soil (T5) - 360 J mL⁻¹, and natural soil (T6) - 576 J mL⁻¹. After dispersion, the material was centrifuged and filtered again to separate the occluded light fraction (OLF). The remaining fraction in the centrifuge tube corresponds to the heavy fraction (HF). The FLF and OLF were dried at 50 °C, ground in an agate mortar and analyzed for C content (TruSpec CHN Elemental analyzer). The C in the HF was computed as the difference between TOC and the light fractions (HF = [TOC - (FLF + OLF)]). The TOC and C stocks in the fractions FLF, OLF and HF were calculated based on the C contents in each fraction and Bd. From these data, the distribution of these fractions was calculated as percentage of the TOC stock.

### Carbon management index estimated from particle-size and density fractions of OM

The carbon management index was estimated from the particle-size and density fractions of OM for each treatment, according to equation 1. The C stocks of the fractions CCF and FLF were used as labile C, while the C stock of the fractions MAC and HF were used as non-labile C. The bare constructed soil (T5) was used as control treatment (CMI = 100), since the natural soil (T6) did not represent the conditions of the experimental area, serving only for demonstrative purposes of the original soil conditions.

\[
\text{CMI} = CPI \times LI \times 100 \quad \text{Eq. 1}
\]

where:

- CPI (Carbon Pool Index) = TOC stock of the treatment/TOC stock of the control treatment (T5);
- LI (Lability index) = L treatment/L control treatment (T5); and,
- L (lability) = stock of labile C (CCF or FLF)/stock of non-labile C (MAC or HF);

### Statistical analysis

The results for T1, T2, T3, and T4 were subjected to analysis of variance and Duncan’s test at 5 %. The results of the treatments T5 and T6 were not included in the statistical analysis for not being part of the experimental design and not representing the soil conditions of the experiment. Therefore, they
were only used as parameter in the degraded soil recovery process.

The Pearson correlation coefficient (r) was calculated as the correlation between CMI estimated from the particle-size and density fractions of OM.

**RESULTS AND DISCUSSION**

**Total carbon stocks and C distribution in physical compartments**

The TOC stock ranged from 4.60 to 5.92 Mg ha⁻¹ and did not differ between treatments (Table 2), possibly because this soil had been constructed only six years before and because the four species used for vegetation were all grasses. Similar TOC contents were reported by Sever and Makineci (2009) in the surface layers (0.00-0.01 and 0.01-0.03 m) of a soil constructed after coal mining and vegetated with *Pinus pinaster* Aiton 17 years earlier.

Compared to the natural soil (T6), it was observed that due to the mining processes and soil construction, the TOC stock in T5 was reduced by 316 %. However, the cover crops induced a partial recovery of TOC stocks in the constructed soil of 79 % (in the mean), compared to T5. On the other hand, these stocks were less than half of those found in the natural soil. Chatterjee et al. (2009) compared the TOC stock in the 0.00-0.10 m layer of soil constructed after coal mining 11 years earlier with a natural soil and observed that the natural soil had 10 Mg ha⁻¹ more TOC than the constructed soil. According to the authors, the low TOC stocks of constructed soils can be attributed to the greater mineralization and leaching of OM and to soil erosion, due to low soil aggregation. Since the average annual rainfall in the experimental area is considerably high (1,400 mm), dissolved organic compounds may have been leached into subsurface layers, which, along with the high degree of soil degradation, may have contributed to the low TOC stock in the constructed soil.

The CCF stock ranged from 1.73 to 2.61 Mg ha⁻¹ and MAC from 2.87 to 3.31 Mg ha⁻¹ (Table 2). However, not even the coarse fraction, usually more sensitive to management changes, was efficient in detecting the effects of the different cover crops on OM composition in the constructed soil.

Although there was no difference among the treatments with regard to the CCF and MAC stocks, their values were on average 166 and 45 % higher, respectively, than those of the bare soil. This reflects the effect of plant biomass on the recovery of C stocks in constructed soil, especially regarding CCF, which is derived from plant and hyphal residues that still have recognizable cell structures. Since no cover crops were used in T5 and taking into account that the MAC stock depends on the amount of organic material that is transferred from the CCF fraction, the lower CCF and MAC stocks in this treatment had been expected. The highest CCF and MAC stock values in T6 can be explained by the constant addition of plant residues to the soil surface over the years and by the absence of disturbance by coal mining and soil construction processes.

The CCF/TOC ratio ranged from 27 to 44 % (Table 2). Due to the small specific surface area and low surface charge density, little or no organic material is associated to the CCF fraction, and its contribution to the maintenance of soil OM is usually lower than that of MAC (Baldock et al., 1992; Christensen, 1992). However, CCF is an important compartment for being an energy source for soil biota and a nutrient source for plants. Additionally, the CCF is often a sensitive indicator, in the short term, of management-induced changes in the soil (Dieckow, 2003). The highest proportion of the TOC stock was represented by MAC (56-73 %) (Table 2). These data corroborate the findings of Conceição (2006), who reported that the proportion of CCF was lower (12-23 %) than that of MAC (77-88 %) in the 0.00-0.05 m layer of an Alfisol under native vegetation and crop rotation in

| Treatment     | TOC   | CCF  | MAC  | CCF/TOC | MAC/TOC |
|---------------|-------|------|------|---------|---------|
| T1 - Hemarthria | 5.92 a | 2.61 a | 3.31 a | 44 | 56 |
| T2 - Paspalum   | 5.10 a | 2.02 a | 3.09 a | 39 | 61 |
| T3 - Cynodon    | 4.60 a | 1.73 a | 2.87 a | 37 | 63 |
| T4 - Brizantha  | 5.37 a | 2.36 a | 3.01 a | 44 | 56 |
| T5 - Bare soil  | 2.93   | 0.82  | 2.11  | 27 | 73 |
| T6 - Natural soil | 12.20 | 4.27 | 7.92 | 35 | 65 |

Means followed by the same letter in the column do not differ by the Duncan test at 5 %.
NT and CT. This fraction contains more humified material associated with the soil mineral matrix, and is a fundamental constituent for stabilization and maintenance of C stocks.

The density fractions were more sensitive than the particle-size fractions to detect the effects of different plant species on soil OM. The C stocks in FLF were highest in the treatments Hemarthria and Brizantha (Table 3), probably due to the better adaptation of these species to the adverse soil and climate conditions in the experimental area. Hemarthria is a plant that grows well in soils with pH 5.5-6.5, tolerates drought, frost and cold, and has a high growth rate and potential of biomass production. In this sense, Brizantha is a fast-growing plant, adapted to clayey soils, develops well in soils with pH 5.0-7.5, has average drought-tolerance and is adapted to low soil fertility. On the other hand, Cynodon and Paspalum have characteristics such as higher fertility requirement and slow growth, consequently, the potential for biomass production of Hemarthria and Brizantha is superior (Santos et al., 2008), resulting in higher C stocks in FLF.

The C stocks in OLF decreased in the sequence T1>T3>T4>T2 (Table 3). In the same experiment, Gonçalves (2008) evaluated the mean weight diameter (MWD) of aggregates in the 0.00-0.05 m layer, with the following results: T1 = 1.46 mm, T3 = 1.27; T4 = 1.25 mm and T2 = 1.15 mm, i.e., the MWD values decreased in the same sequence as observed in this study for C stocks in the OLF. According to the author, the highest MWD in T1 is a result of the higher OM input and greater cover of the soil surface. Consequently, the highest C stock in OLF of T1 could be explained by the enhanced physical protection of OM within the aggregates. Conceição (2006) observed a high correlation (r = 0.97) between C in the OLF and the MWD in aggregates of the 0.00-0.05 m layer of an Alfisol under different cropping systems. According to the author, this reinforces the important role of plants in the formation of soil aggregates and consequently in C occlusion, where the organic material served as nucleus for aggregate formation and their decomposition products as aggregate stabilizers, decreasing the access of microorganisms and their enzymes to the organic compounds.

The C stocks in HF (2.75-3.64 Mg ha⁻¹) did not differ between treatments (Table 3). This fraction consists of organic material in an advanced stage of decomposition, which is associated to the mineral matrix of the soil, in particular to clay, and is characterized by being the most stable density fraction with the lowest cycling rate.

The C stocks in FLF, OLF and HF were lowest in T5, opposite to T6, in which the stocks were highest (Table 3), proving C losses in all OM fractions as a result of coal mining and soil construction processes.

In general, the contribution of density fractions to TOC decreased in the sequence HF (67 %)>FLF (19 %)>OLF (14 %). These data differ from those reported by Conceição et al. (2008) and Rosa (2011), in the surface layer of an Alfisol and an Albaqualf, respectively, where the proportion of OM fractions decreased HF>OLF>FLF. These authors explained their results based on the protection mechanisms of each fraction. In FLF, only chemical recalcitrance is responsible for OM stability, while in OLF, OM occlusion within the aggregates also contributes to OM preservation. In HF, aside from the two above mechanisms, the interaction of OM with the mineral fraction of the soil is also influential. For these reasons, HF and OLF have a slower cycling rate than FLF. In this study, the lower representation of OLF compared to FLF, in general, can be explained by physical deterioration that occurs during coal extraction and soil construction, where aggregates are disrupted, exposing the previously protected OM to decomposition. In the natural soil (T6), the lower representation of OLF than FLF may be due to a higher residue input on the surface, increasing the FLF fraction and because the sand content in the natural soil is high (483.6 g kg⁻¹) in the 0.00-0.05 m layer (Table 1), making aggregation and consequently OM occlusion less efficient.

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Table 3. Carbon stocks in the free light (FLF), occluded light (OLF) and heavy fractions (HF) and FLF/TOC, OLF/TOC and HF/TOC ratios in the 0.00-0.05 m layer of a constructed soil under different cover crops, a bare constructed soil and a natural soil in a coal mining area

| Treatment         | FLF  | OLF  | HF   | FLF/TOC | OLF/TOC | HF/TOC |
|-------------------|------|------|------|---------|---------|--------|
| T1 - Hemarthria   | 1.22 | 1.09 | 3.61 | 21      | 18      | 61     |
| T2 - Paspalum     | 0.86 | 0.61 | 3.64 | 17      | 12      | 71     |
| T3 - Cynodon      | 0.83 | 1.02 | 2.75 | 18      | 22      | 60     |
| T4 - Brizantha    | 1.27 | 0.86 | 3.25 | 24      | 16      | 60     |
| T5 - Bare soil    | 0.31 | 0.69 | 1.88 | 11      | 2       | 87     |
| T6 - Natural soil | 2.65 | 1.87 | 5.50 | 22      | 15      | 63     |

Means followed by the same letter in the column do not differ by the Duncan test at 5 %.
**Plant effect on OM lability and CMI**

The cover crops influenced CPI positively, compared to bare soil. However, these values were lower than those of the natural soil (Table 4), reflecting the increase in soil quality and indicating that the constructed and vegetated soil is at the beginning of the recovery process. The greater OM lability (L) after six years under cover crops compared to T5 was evidenced by both particle-size and density fractionation of OM (Table 4). The labile fractions play a key role in the process of soil quality recovery, for serving as nutrient source for plants and as energy source for microorganisms.

The LI values obtained by density fractionation (1.98 - 3.38) were higher than the values obtained by particle-size fractionation (1.65 - 2.19) (Table 4). Due to the absence of cover crops in the control treatment and OM division into three different fractions in the density fractionation, in proportion to T5, there was greater efficiency in obtaining the FLF fraction than in obtaining the CCF fraction, justifying the LI results.

Regardless of the fractionation type, LI was higher in all treatments in comparison to T5 (LI = 1.00), with highest values of the cover crops Hemarthria and Brizantha (Table 4), reinforcing the greater potential of these species for the recovery of soil quality. Comparing the effect of different tillage systems in the 0.00-0.20 m layer of an Alfisol, Conceição et al. (2014) also reported higher LI values obtained by density fractionation (sieved soil <2 mm) than by particle-size fractionation. According to the authors, this was due to the fact that the C considered labile in the particle-size fractionation was that retained on a 0.053 mm sieve, but for the density fractionation, that retained in a 0.0014 mm filter, resulting in a higher yield of the FLF fraction in relation to the CCF fraction, i.e., the density fractionation included possible organic particles between 0.0014 and 0.053 mm in the labile C classification.

Similarly as for LI, regardless of the fractionation method, the CMI was higher in all treatments than in T5 (CMI = 100), with values ranging from 249 to 427 by particle-size fractionation, and 343 to 612 by density fractionation (Table 4). This was due to the higher efficiency (in proportion to T5) in the separation of FLF in comparison to the CCF fraction, resulting in increased LI and consequently in a higher CMI. Of the cover crops, those with highest CMI were Hemarthria and Brizantha, probably due to a better adaptation of these species to the environment, providing greater biomass input, greater protection against soil erosion and especially greater C accumulation in the labile OM fraction. Despite the apparent adaptation of these species to the adverse climate and soil conditions, no treatment exceeded the CMI of natural soil (577 by particle-size fractionation and 1,204 by density fractionation), reflecting the high degradation degree and poor quality of constructed soil. Lima et al. (2012) evaluated soil physical properties such as compressibility and limiting water range in the same experiment and also observed greater potential for recovery of constructed soil of the species Brizantha and particularly of Hemarthria than of Paspalum and Cynodon.

The CMI estimated by the particle-size fractions was highly correlated with CMI estimated from OM density fractions (Figure 1). However, density fractionation resulted in a greater range of CMI values, because in relation to T5, the efficiency in obtaining the FLF fraction was greater than for the CCF fraction, and consequently the estimated CMI data were higher. These data corroborate those of Conceição et al. (2014). In the 0.00-0.20 m layer of an Alfisol under NT, the authors found a high correlation (r = 0.92, p<0.01) between particle-size CMI and density CMI. However, the authors reported significantly lower CMI values (maximum 140) in comparison to our data, which can be attributed to the use of a native field as control treatment to calculate CMI, whereas in this study a negative control (bare constructed soil) was used.

**Table 4. Carbon pool index (CPI), carbon lability (L), lability index (LI) and carbon management index (CMI), obtained by physical particle-size and density fractionation of organic matter in the 0.00-0.03 m layer of a constructed soil under different cover crops, a bare constructed soil and a natural soil in a coal mining area**

| Treatment       | CPI | Particle-size fractionation | Density fractionation |
|-----------------|-----|----------------------------|----------------------|
|                 |     | L  | LI | CMI |     | L  | LI | CMI |
| T1 - Hemarthria | 2.02| 0.81| 2.19| 427 | 0.35| 2.87| 565 |
| T2 - Paspalum   | 1.74| 0.64| 1.74| 299 | 0.24| 1.98| 343 |
| T3 - Cynodon    | 1.57| 0.61| 1.65| 249 | 0.30| 2.46| 391 |
| T4 - Brizantha  | 1.83| 0.80| 2.15| 373 | 0.41| 3.38| 612 |
| T5 - Bare soil  | 1.00| 0.48| 1.00| 100 | 0.12| 1.00| 100 |
| T6 - Natural soil| 4.16| 0.55| 1.44| 577 | 0.36| 2.94| 1,204 |

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After six years of cultivation, the different cover crops promoted a partial recovery of the total organic carbon stock in the constructed soil, compared to natural unmined soil. The density fractionation was more sensitive than the particle-size fractionation to detect the effects of different cover crops on soil organic matter. The free light and heavy fractions were more appropriate for estimating the carbon management index than the coarse and mineral-associated carbon fractions. Due to the higher recovery of carbon stocks in the free light and occluded light fractions and due to the higher carbon management index, the cover crops Hemarthria and Brizantha are recommended for the recovery of organic matter of the constructed soil.

CONCLUSIONS

After six years of cultivation, the different cover crops promoted a partial recovery of the total organic carbon stock in the constructed soil, compared to natural unmined soil.

The density fractionation was more sensitive than the particle-size fractionation to detect the effects of different cover crops on soil organic matter. The free light and heavy fractions were more appropriate for estimating the carbon management index than the coarse and mineral-associated carbon fractions.

Due to the higher recovery of carbon stocks in the free light and occluded light fractions and due to the higher carbon management index, the cover crops Hemarthria and Brizantha are recommended for the recovery of organic matter of the constructed soil.

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