



SOIL STRUCTURAL AND CHEMICAL CONDITIONS AFTER DISPOSAL OF FABRIC DYE ON A LOAMY SAND SOIL IN SOUTHWESTERN NIGERIA

Fadare, F. J., Salako, F. K., and Adesodun, J. K.¹

Department of Soil Science and Land Management, Federal University of Agriculture, P. M. B. 2240, Abeokuta 110001, Ogun-State, Nigeria.

**Corresponding Author: E-mail address adesodunjk@unaab.edu.ng; jadesodun@yahoo.com):
Tel: +2348033469381*

ABSTRACT

The indiscriminate disposition of dye waste-water from the age long “Adire” industry of Abeokuta, southwest Nigeria is a major concern as the movement of such contaminants in the soil needs to be monitored. This study investigated the effect of fabric dye on soil physical and chemical properties. Treatments were local concentration, spent local-concentration, half local-concentration and twice local-concentration of the dye; and control (no contaminant). Sixteen litres of the dye concentrations were applied on field plots every two days to maintain the soil at 10% field capacity for 3 months. Soil samples were collected from 0-10 cm, 10-20 cm and 20-30 cm depths at termination of application of dyes (3 months) and 12 months after the application of treatments. Results show that macroaggregates (>0.25 mm) were dominant in the control while microaggregates (<0.25 mm) were more in dye polluted plots at 3 months. The mean-weight diameter (MWD) for control was about two times higher (1.73 mm) than that of the treated plots (0.77-0.87 mm). Twelve months after the termination of dye application, macroaggregates (5-2 mm) and microaggregates (<0.25 mm) had similar distributions for the control and treated plots (9.70-25.44 mm). Bulk density for treated plots (1.99-2.10 g/cm³) were significantly ($p < 0.05$) higher than control (1.83 g/cm³). Saturated hydraulic conductivity (Ks) was higher in control (12.6 cm/hr) than treated plots (0.6-10.2 cm/hr). Infiltration rate declined in dye treated plots (2.33- 30.53 cm/hr) at -2 cm water head compared to control (88.00 cm/hr); whereas, at -1 cm water head it ranged from 1.13-23.60 cm/hr in treated plots compared to control (71.87 cm/hr). Sorptivity at -2 cm and -1 cm water head (2.94 cm/hr^{1/2} and 1.60 cm/hr^{1/2}) for control was significantly higher than dye plots (0.00003-0.00006 mm/hr^{1/2}). Exchangeable acidity was significantly higher in the control (6.02-6.17 cmol/kg) than 0.37-0.77 cmol/kg observed in dye plots. However, Na⁺ (21.53-90.23 cmol/kg), phosphorus (13.68-39.92 mg/kg), K⁺ (20-49.67 cmol/kg), Mg²⁺ (97.57-100.63 cmol/kg) and sulphur (4.67-8.93 mg/kg) contents were significantly higher with dye treatment. The pH was more acidic in contaminated plots (6.13-6.53) than near neutral (6.97- 7.00) for control. In conclusion, significant physical and chemical degradation was observed following treatment with the dye and its waste-water indicating negative impact on the soil.

Keywords: Fabric dye; soil degradation; structural and chemical properties

INTRODUCTION

A dye is generally described as a colored substance that has affinity to the substance to which it is applied (Kirk and Othmer, 1980). Classification of dyes is based on how they are used in the dyeing process which includes acid, basic, sulphur, direct or substantive, mordant, vat, reactive and disperse dyes (Kirk and Othmer, 1980). Generally, improper disposal of large quantity of industrial waste may cause contamination of air (via volatilization and fugitive dust emission), surface water (from surface runoff or over land flow and groundwater seepage), ground water (through leaching/ infiltration), soils (due to erosion, including fugitive dust generation/ deposition and tracking), sediments (from surface runoff/ overland flow seepage and leaching), and biota due to biological uptake and bioaccumulation (Virendra and Pandey, 2005). The commonly used dye by the *adire* industry of Abeokuta is the sulfur or sulfide dyes which contains sulfur or are precipitated from sodium sulfide bath, formic acid, caustic soda, metallic salt and sodium nitrate. When dye wastes accumulate in the soil, it increases the biochemical oxygen demand (BOD) of soils and soil organisms (Lorimer et al., 2001). It does appear that disposition of dye wastes will lead to introduction of other contaminants into soil and water, given their compositions. Some components are known to cause extreme acidity (e.g. sulphur) or extreme alkalinity and destruction of soil structure (e.g., sodium) while emissions from the dyes (e.g., nitrous oxide) could contribute to greenhouse warming. Therefore, it is important to understand the movement of such contaminated water in the soil and possible uptake by crops if used for irrigation purpose. The objective of this study was to determine the effect of the dye on some soil physical and chemical properties.

MATERIALS AND METHODS

Experimental site

The study area was located in the Federal University of Agriculture, Abeokuta (Lat. 7.12⁰ N and Long. 3.23⁰ E) Ogun State, Nigeria. The rainfall distribution for this area is bimodal with wet season from March to October and dry season from November to February. The mean annual rainfall is about 1400 mm with the maximum in July. The mean annual minimum and maximum temperature are 22.2⁰C and 33.3⁰C respectively. The experiment was carried out in the dry season to minimize much rainfall impact on data collected. The particle size distribution of the project site is shown in Table 1.

Field Experiment and sampling

A land area of 204.74 m² was cleared manually with each plot measuring 2.25 m² (1.5m x1.5m) and a separating distance of 3 m between and along each plot. A basal dose of NPK was added to each plot. The treatments on these plots were control (no contaminant), local-dye concentration, spent local-dye concentration, half local-dye concentration and twice local-dye concentration which were arranged in a randomized complete block design (RCBD) and replicated three times. A local-dye concentration as used in this study refers to the concentration of fresh dye typically used in Itoku Adire Fabric Industry in Abeokuta, Nigeria. The local-dye concentration used for one piece of cloth (14.6 m²) is a mixture of six spoonful of sulphur dye, two spoonful of Na(OH)₂, and two spoonful of Na₂S. Chemical composition of the local-dye is shown in Table 2. The spent local-dye concentration represented dye wastewater from the fabric processing industries which are normally disposed of to the environment.

The soil was watered to field capacity (FC, -10 kPa) using the equation of Salako et al. (2006) to calculate the required amount of water:

$$FC = 1.01 - (1.04 \times 10^{-3} \text{Total Sand}) - 5.88 \times 10^{-4} \text{Clay} \quad (1.0)$$

Water required for each sub-plot at field capacity was 150 litres. Thus, water required at 10% capacity was approximately 16 litres. Therefore, 16 litres of dye concentration was applied to each sub plot every 2 days with watering can. Soil samples were taken at 0-10, 10-20 and 20-30 cm before the treatment application, at 3 months after application of dyes and 12 months after stoppage of application.

LABORATORY STUDIES

Physical properties

The soil samples taken at 0-20 cm depth were air-dried at room temperature and then pre-sieved through a 5.00 mm sieve. Clods greater than 5.00 mm were crushed by hand (along lines of natural cleavage) to pass through the sieve. The distribution of aggregates was estimated by wet-sieving technique which was carried out manually by regular lifting and suspension of a nest of sieves in a bucket of water with 50 cm height and 20 cm diameter as described by Sarrantonio (1991). In this procedure, 50 g of less than 5 mm aggregates were placed in the topmost of a nest of sieve of mesh diameter 2, 1, 0.5, 0.25 and < 0.25 mm and pre-soaked in water for 10 minutes before lifting up and down in water 20 times. The resultant aggregates on each sieve were dried at 105°C for 24 hours before their masses were recorded. The mass of less than 0.25 mm aggregates were obtained by difference. The percentage water stable aggregates (WSA%) in each of the following size ranges 5-2, 2-1, 1-0.5, 0.5-0.25 and <0.25 mm were determined using the method of Van Bavel as modified by Kemper and Rosenau (1986), while aggregate stability was expressed as:

$$MWD = \sum_{i=1}^n X_i W_i \quad (2)$$

where *MWD* is the mean weight diameter of wet-stable aggregates (mm), X_i the mean diameter of each size fraction (mm) and W_i the

proportion of the total sample weight (WSA) in the corresponding size fraction.

Soil bulk density was determined with core samples. Saturated hydraulic conductivity (K_s) was determined by constant head soil core method of Reynolds (1993) by transposed Darcy's equation for vertical flow of liquid:

$$K_s = [V (Z)] / A (t) (H)$$

where V is the volume of water (L^3) that flowed through a cross-sectional area (L^2) in time t (T), and H is the hydraulic head difference (L) imposed across the sample length Z (L).

Water infiltration into the soil was measured using the CSIRO (1988) Disc permeameter before the application of treatment and immediately after the stoppage of treatment application. The permeameter was used to measure unsaturated and saturated flow.

For unsaturated flow, infiltration measurement was carried out on an even undisturbed surface prepared for the placement of about 1 cm thick diatomaceous earth within a 20 cm ring. The diatomaceous earth was thus the contact material between the soil and the disc permeameter. Infiltration measurement at -2cm and -1cm water heads started immediately the water from the chamber of the permeameter was allowed to flow into the soil. Time taken for measured depth of water flow as indicated on the graduated chamber was recorded with the aid of a stopwatch. Soil samples were taken before and after measurements for gravimetric water content.

The saturated flow set-up was different from the unsaturated flow because passage of water into soil was from bigger openings of wire mesh, unlike the fine membrane for water passage under unsaturated flow. Also a positive water head of 1 cm was maintained for saturated flow. As soon as flow started from the water chamber, a stopwatch record was taken along the changing height of water

in the chamber. Soil samples were also taken before and after measurements for gravimetric water content.

Cumulative infiltration (CI) for both unsaturated and saturated flow was calculated as presented by CSIRO (1988):

$$Q/r^2 = (SR - SR_1) (RC)/ r^2 \quad (3)$$

where SR is the scale reading at the time of measurement, SR_1 is the initial scale reading, and RC is the reservoir calibration (17.7 cm³/cm), and area (r^2) = 314.2 cm².

The steady-state flow rate (q/r^2) was obtained by plotting the CI during the last part of the infiltration run as a function of time (t). Thus,

$$q/r^2 = (\text{Scale increment}) (RC)/ (\text{Area}) (\text{Average time}) \quad (4)$$

The Sorptivity (S) was calculated from the plot of Q/r^2 against the square root of time ($t^{1/2}$); while the slope of the straight line portion is the sorptivity in length/ time^{1/2}.

Chemical properties

Soil samples collected at 0-10, 10-20, and 20-30 cm depth were air-dried and sieved through a 2 mm sieve. 5g of each sieved soil was used for chemical analysis. Total acidity (H^+ and Al^{3+}) was determined by titration with 0.05 N NaOH. Available sulphur was determined. Ca ($(H_2PO_4)_2$ extractant). The amount of sulphate-sulphur was determined turbidimetrically as $BaSO_4$. Available phosphorus was determined by Bray I method. Exchangeable bases were by ammonium acetate replacement method. This procedure involved addition of 1 ml strontium nitrate solution into 25 ml of the leachates. Ca and Mg were measured with atomic absorption spectrophotometry (AAS), while K and Na were measured with a flame photometer.

DATA ANALYSIS

The data collected were analyzed using the analysis of variance (ANOVA) procedure of SAS (2003), while significant treatment means were separated using LSD at 5%.

RESULTS

Soil aggregation and stability

Macroaggregates (> 0.25 mm) were more dominant in the control than in soils treated with dye at 3 months after the application (Table 3). Invariably, there were more microaggregates (< 0.25 mm) with dye treatment than the control. All the dye treatments were generally similar in aggregate size distribution. The mean weight diameter (MWD) for the treated plots ranged from 0.77 - 0.87 mm while that of the control was about two times higher than that of the treated soils (Table 3).

At twelve months after the termination of dye application, the distribution of 5-2 mm macroaggregate fraction and microaggregates (< 0.25 mm) were similar for the control and plots treated with dye (Table 4). Also, MWD ranged between 1.41 and 1.73 mm. Application of half local dye concentration significantly ($p < 0.05$) reduced the MWD (1.41 mm) compared to control (1.73 mm). The general trend showed reduction in stability of this soil with application of the fabric dye of different concentrations.

Soil bulk density and saturated hydraulic conductivity

Three months after the application of dye, soil bulk density of the treated plots were significantly ($p < 0.05$) higher than that of the control (Table 5). At 12 months following the stoppage of dye application, bulk density in plot treated with half concentration of dye was similar to the control; whereas plots treated with other concentrations of dye had significantly ($p < 0.05$) higher bulk density.

Treatment with different dye concentrations significantly ($p < 0.05$) reduced the saturated

hydraulic conductivity (K_s) compared to control (12.6 cm/hr). Mean K_s was least (0.6 cm/hr) in plots treated with twice-local dye concentration; and this was followed by 2.4 cm/hr observed for spent-local dye concentration (Table 5).

The K_s is an indicator of soil's ability to imbibe and transmit plant-available water to the root zone, as well as drain excess water out of the root zone (Reynolds et al., 2007). Since K_s value in the range of 1.8 cm/hr to 18 cm/hr may be considered "ideal" for promoting rapid infiltration and redistribution of needed crop-available water, reduce surface runoff and erosion, and rapid drainage of excess soil water; moderately rapid K_s (12.6 cm/hr) observed in plot with no dye (control) and slow K_s induced by application of the fabric dye and its waste indicated deleterious effect of dye on soil properties and function.

Infiltration parameters

Cumulative infiltration (CI) and steady state infiltration rates referred to in this study as steady state flow rate (SFR) and sorptivity (S) measured from the plots before and after application of the dyes are presented in Tables 6, 7 and 8. These infiltration parameters were measured at -2 cm and -1 cm water head for unsaturated flow, while a positive 1 cm water head was maintained for saturated flow.

Saturated and unsaturated water flows were similar for all the plots before treatment with different fabric dye concentrations. However, infiltration measurements 3 months after application of different dye concentrations significantly ($p < 0.05$) decreased water flow through this soil over the control. Cumulative infiltration decreased significantly to 67% and 82% with twice-local dye and half-local dye concentrations respectively; whereas, reduction in saturated cumulative infiltration was about 100% in plots treated with the different dye concentrations (Table 6). This trend was observed for steady state flow rates (Table 7).

Soil water sorptivity (S) was also negatively affected with application of the different dye concentration (Table 8). Lower S observed in plots treated with dye was an indication of decline in infiltration rates. Generally, there was 100% decrease in sorptivity with dye treatment compared with control which was 5.71 mm hr^{1/2} and 1.60 mm hr^{1/2} for saturated and unsaturated condition respectively. This observation revealed negative impact of indiscriminate disposal of waste fabric dye on the environment with resultant soil surface sealing.

Effects of dye application on soil chemical properties

Exchangeable acidity at different depths (Table 9) was significantly ($p < 0.05$) higher in control than in plots treated with the fabric dyes. At 3 months, soil acidity was reduced by 94% and 87% in plots treated with local-concentration and spent local-concentration respectively within 0-10 cm depth. The trend observed at lower depths, i.e. 10-20 cm and 20-30 cm, at 12 months after application of the dyes was similar to that observed at 3 months. However, sodium (Na^+) and available phosphorus (P) contents of the soil were significantly increased by dye treatment compared to control (Table 10 and 11). Highest concentrations of these elements were observed in plots treated with local- and twice-local concentration of dye. Reduction over 12 months for sodium was averagely 45% for the simulated local dye concentration, 28% for the spent dye, 39% for half local concentration and 18% for twice the local concentration (Table 10).

Potassium (K^+), magnesium (Mg^{2+}) and sulphur (S) levels were also significantly ($p < 0.05$) lower in control plot than in plots treated with different dye concentrations (Table 12, 13 and 14). Irrespective of soil depth and sampling period, contents of K^+ and Mg^{2+} varied in plots treated with dye while sulphur level was highest in plots treated with twice local-concentration of the dye. Soil pH values

were slightly acidic with dye treatment compared to neutral pH observed in control (Table 15).

DISCUSSION

The dye used for the Adire fabrics at Itoku in Abeokuta of southwest Nigeria has a very high concentration of sodium ion (Table 2). The concentration of sodium in waste water containing spent dye was 3.5 times that of fresh dye. It appears, therefore, that Na^+ content was increased during usage, perhaps, by some other additives during dyeing of the fabrics. Sodium is a soil dispersive agent, and it is commonly used to separate soil particles effectively in evaluating particle size distribution. Furthermore, saline, sodic or alkaline soils with high contents of Na^+ are often described as 'structureless' because of the dispersed state of soil particles, which clog soil pores and create massive structures. The low content of Na^+ in the effluent (Table 2) also indicates that with proper dilution, Na^+ content could be effectively reduced to 7 times less the fresh dye concentration and 23 times less the spent dye concentration. Phosphorous and sulfur were relatively low in the dyes.

The dispersive nature of the dye on soil was reflected with higher percentage of aggregates < 0.25 mm in the dye application treatments than the control 3 months after application (Table 3). Soil macroaggregates were weakened and dispersed with addition of dye. Thus, aggregate stability of the control plot was higher than dye application plots. However, the effects of the dye had been removed 12 months after application (Table 4) as indicated by soil aggregate stability being restored to the pre-trial level. This must have been due to leaching of the Na^+ soil by the rains. Furthermore, this deduction is consistent with the observation from effluents from the dye industry which contained far less amount of Na^+ due to dilution effect (Tables 2 and 3). Therefore, the results showed that the soil recovered in terms of aggregate stability from deleterious effect of dye from the Itoku dye

industry 12 months after stopping treatment with dye.

Further proof of the deleterious effect of the dye was shown by the soil bulk density after 3 months of dye application, when all dye treated plots had higher bulk density lower water transmission. The higher saturated hydraulic conductivity of the control indicates that pores of the control plot were conducting water more effectively than the dye treated plots. Furthermore, the plot with half of local fresh dye concentration conducted water more effectively in plots treated with other dye concentrations.

Bulk density values observed at 12 months after application of the dye suggested limited recovery of soil structure from the deleterious effect of the dye. Although exchangeable acidity was significantly higher and Na^+ was significantly lower in the control plot than the dye application plot, the magnitudes of the differences for sodium was more pronounced than exchangeable acidity. The extremely high contents of Na^+ sustained for over 12 months, this suggest that soil structure for the dye application plots could not have reliably recovered. Thus, higher concentration of Na^+ in the dye waste-water (Table 2) resulted in a reduced potential for removal from the soil after application. This again suggested that leaching of Na^+ between the beginning of the experiment and 12 months after stoppage of application of dyes by rain could not be expected to be a remedial solution. There might be need for gypsum application, in remediation of these soils, on a short-term basis; otherwise prolonged fallow must be allowed to allow salts to be leached.

However, substantial amounts of cations were also added to the soil through the various dye concentrations applied. Therefore, application of gypsum under the sub-humid environment with more than 1000 mm of rainfall might not be necessary, as persistent rains would wash off sodium and could leave the exchange site

to the other cations. The problem that could again arise might be from sulphur addition to soil by the dyes. This could promote acidification after elimination of sodium. Invariably, the addition of the dyes in this ecosystem could create a complex management problem to restore the soil to a productive state.

The dye waste-water could neither be used for irrigation of crops nor disposed in areas with natural vegetation; therefore, crops and plants would not grow because of the unfavorable structural and chemical conditions created by the application of the dye waste-water. Based on the data generated it was observed that fabric dye causes a significant level of soil physical and chemical degradation in the soil at the termination of the experiment causing a significant drop in the infiltration rate of the soil as water was seeping off the soil surface. The chemical degradation observed resulted in significant increase in salinization and alkalinization of this soil.

CONCLUSION

Soil physical quality is central concept for quantifying land degradation and soil structure. Measures of water infiltration may give better indication of soil physical status and suitable structural status for biological processes of root development and is largely

dependent on the relationship of water and air-filled-pores in soil.

The study was conducted to assess the effect of dye waste-water on soil structural and chemical quality. The major findings and conclusion of this study are as follows:

1. Fabric dye and dye waste-water are toxic and it seals the macro and micro pores in the soil thereby preventing air and water exchange in the soil.
2. Ponding of dye on the soil forms surface seals and crusts which prevent water and air from entering the soil due to the dispersing of the soil particles causing surface sealing.
3. The soil structure has been greatly damaged as the chemical composition of dye and dye waste-water caused potential salinizaion and alkalinization of the soil.
4. A significant level of soil physical degradation occurred leading to a significant drop in the infiltration rate of the soil as a result of significant salt buildup in the soil.
5. Indiscriminate dumping of dye waste-water on soil could result in leaching hazardous elements deep into the soil water table, thereby releasing the toxic components into ground water which may flow into nearby stream and river thereby enhancing adverse effects on the aquatic lives.

Table 1: Particle size distribution of the experimental site

| Sampling depth (cm) | % Sand | % Clay | % Silt |
|---------------------|--------|--------|--------|
| 0-10 | 78.4 | 20.0 | 1.60 |
| 10-20 | 78.4 | 20.0 | 1.60 |
| 20-30 | 69.2 | 24.0 | 6.80 |

Table 2: Some chemical composition of the local-dye

| Sample | Na ⁺ (mg/kg) | P (mg/kg) | S (mg/kg) |
|-----------------------|-------------------------|-----------|-----------|
| Dye ¹ | 538 | 78.113 | 19.528 |
| Used Dye ² | 1898 | 0.038 | 0.004 |

¹ Dye refers to researcher-mixed equivalent of local concentration or fresh dye.

² Used or spent dye refers to dye already used (wastewater) by the fabric industry.

Table 3: Percentage water-stable aggregates and mean weight diameter (mm) after 3 months of application of dyes

| Treatment | Aggregate size (mm) | | | | | MWD |
|-------------------|---------------------|---------|----------|-----------|--------|-------|
| | 5.0-2.0 | 2.0-1.0 | 1.0-0.50 | 0.50-0.25 | <0.25 | |
| Control | 31.00a | 22.07a | 31.77a | 6.33b | 10.64b | 1.73a |
| Local conc. | 10.73b | 14.37b | 18.70b | 16.41a | 39.44a | 0.84b |
| Spent local cont. | 9.03cd | 12.43b | 25.47ab | 13.81a | 33.97a | 0.79b |
| Half local conc. | 8.17d | 13.57b | 20.53b | 19.07a | 39.57a | 0.77b |
| Twice local conc. | 9.77bc | 15.67b | 26.03ab | 14.57a | 33.97a | 0.87b |

In columns, different letters indicate significant difference at 5% level

Table 4: Percentage water-stable aggregates and mean weight diameter (mm), 12 months after application of dye treatments

| Treatment | Aggregate size (mm) | | | | | MWD |
|-------------------|---------------------|---------|----------|-----------|--------|--------|
| | 5.0-2.0 | 2.0-1.0 | 1.0-0.50 | 0.50-0.25 | <0.25 | |
| Control | 90.97a | 22.04a | 33.77a | 6.34c | 10.64a | 1.73a |
| Local conc. | 31.67a | 15.74d | 23.30b | 12.80ab | 16.37a | 1.59ab |
| Spent local cont. | 31.33a | 17.07cd | 22.43b | 15.34a | 13.93a | 1.60ab |
| Half local conc. | 24.80a | 19.00bc | 25.44b | 11.40b | 19.33a | 1.41b |
| Twice local conc. | 26.24a | 19.97ab | 25.70b | 9.70bc | 18.70a | 1.47ab |

In columns, different letters indicate significant difference at 5% level

Table 5: Bulk density and saturated hydraulic conductivity of the soil at 5cm depth after 3 months of dye application and bulk density at 12 months after stoppage of dye application

| Treatment | Bulk density (g/cm ³) after 3 months | Bulk density (g/cm ³) after 12 months | Saturated hydraulic conductivity (cm/hr) after 3 months |
|-------------------|---|--|---|
| Control | 1.85c | 1.80d | 12.6a |
| Local conc. | 2.03ab | 1.89bc | 4.2c |
| Spent local conc. | 2.08a | 2.00a | 2.4cd |
| Half local conc. | 1.99b | 1.85cd | 10.2b |
| Twice local conc. | 2.10a | 1.93b | 0.6d |

In columns, different letters indicate significant difference at 5% level

Table 6: Infiltration rate (cm/hr) after 30 minutes before and 3 months after dye application

| Treatment | Before Dye Application | | | Three Months After Dye Application | | |
|-------------------|------------------------|---------|-----------|------------------------------------|--------|-----------|
| | Unsaturated flow | | Saturated | Unsaturated flow | | Saturated |
| | -2 cm | -1 cm | 1 cm | -2 cm | -1 cm | 1 cm |
| Control | 129.47b | 103.00a | 6880.0a | 88.00a | 71.87a | 3016.8a |
| Local conc. | 139.20ab | 98.27a | 6840.0a | 8.73c | 1.13d | 1.8b |
| Spent local conc. | 136.73ab | 111.87a | 6600.0a | 2.33c | 1.67d | 2.0b |
| Half local conc. | 148.33ab | 123.73a | 6480.0a | 22.07b | 13.00c | 5.9b |
| Twice local conc. | 129.47b | 125.87a | 6720.0a | 30.53b | 23.60b | 9.4b |

In columns, different letters indicate significant difference at 5% level.

Table 7: Cumulative infiltration (cm) after 60 minutes before and 3 months after dye application

| Treatment | Before Dye Application | | | Three Months After Dye Application | | |
|-------------------|------------------------|-------|-----------|------------------------------------|-------|-----------|
| | Unsaturated flow | | Saturated | Unsaturated flow | | Saturated |
| | -2 cm | -1 cm | 1 cm | -2 cm | -1 cm | 1 cm |
| Control | 3.65b | 2.93a | 182.52a | 2.48a | 2.02a | 131.51a |
| Local conc. | 3.93ab | 2.76a | 192.66a | 0.25c | 0.03d | 0.05b |
| Spent local conc. | 3.85ab | 3.11a | 185.23a | 0.07c | 0.05d | 0.06b |
| Half local conc. | 4.18ab | 3.48a | 182.52a | 0.62b | 0.37c | 0.17b |
| Twice local conc. | 4.48a | 3.55a | 189.28a | 0.72b | 0.67b | 0.27b |

In columns, different letters indicate significant difference at 5% level.

Table 8: Steady state flow rate (cm/hr) after 80 minutes before and 3 months after dye application

| Treatment | Before Dye Application | | | Three Months After Dye Application | | |
|-------------------|------------------------|--------|-----------|------------------------------------|-------|-----------|
| | Unsaturated flow | | Saturated | Unsaturated flow | | Saturated |
| | -2 cm | -1 cm | 1 cm | -2 cm | -1 cm | 1 cm |
| Control | 8.43a | 6.19ab | 15.52a | 8.09a | 6.28a | 14.28a |
| Local conc. | 9.11a | 6.86a | 12.93a | 1.01cb | 0.45b | 0.34b |
| Spent local conc. | 7.98a | 5.73ab | 14.73a | 1.12cb | 0.56b | 0.34b |
| Half local conc. | 7.31a | 5.01b | 14.06a | 1.57b | 0.45b | 0.34b |
| Twice local conc. | 7.76a | 5.62ab | 13.61a | 0.89c | 0.34b | 0.34b |

In columns, different letters indicate significant difference at 5% level.

Table 9: Exchangeable acidity (cmol/kg) at 3 Months and 12 Months after dye application

| Treatment | At 3 months | | | At 12 Months | | |
|-------------------|-------------|---------|---------|--------------|---------|---------|
| | 0-10cm | 10-20cm | 20-30cm | 0-10cm | 10-20cm | 20-30cm |
| Control | 6.02a | 6.12a | 6.17a | 6.02a | 6.12a | 6.18a |
| Local conc. | 0.37d | 0.50bc | 0.50b | 0.47b | 0.43c | 0.53c |
| Spent local conc. | 0.77b | 0.67b | 3.57ba | 0.70b | 0.68bc | 0.52c |
| Half local conc. | 0.50c | 0.53bc | 0.60b | 0.43b | 0.57bc | 0.47c |
| Twice local conc. | 0.47cd | 0.43c | 0.43b | 0.70b | 0.77b | 0.77b |

In columns, different letters indicate significant difference at 5% level.

Table 10: Soil sodium (cmol/kg) at 3 Months and 12 Months after dye application

| Treatment | At 3 months | | | At 12 Months | | |
|-------------------|-------------|---------|---------|--------------|---------|---------|
| | 0-10cm | 10-20cm | 20-30cm | 0-10cm | 10-20cm | 20-30cm |
| Control | 0.21c | 0.23c | 0.22c | 0.28c | 0.26c | 0.25c |
| Local conc. | 75.33ab | 65.87a | 56.20a | 32.47b | 38.80ab | 35.00b |
| Spent local conc. | 47.40c | 39.07b | 30.27b | 22.53b | 24.20b | 32.53b |
| Half local conc. | 52.03bc | 38.93b | 33.27b | 27.37b | 21.53b | 24.87b |
| Twice local conc. | 90.23a | 72.70a | 64.50a | 59.00a | 59.33a | 63.87a |

In columns, different letters indicate significant difference at 5% level.

Table 11: Available phosphorus (mg/kg) at 3 Months and 12 Months after dye application

| Treatment | At 3 months | | | At 12 Months | | |
|-------------------|-------------|---------|---------|--------------|---------|---------|
| | 0-10cm | 10-20cm | 20-30cm | 0-10cm | 10-20cm | 20-30cm |
| Control | 3.64d | 3.50d | 3.68d | 3.57d | 3.64d | 3.71d |
| Local conc. | 17.80b | 17.70b | 17.65b | 16.67b | 17.00b | 17.02b |
| Spent local conc. | 15.61c | 16.07bc | 16.04bc | 15.33bc | 15.89bc | 15.96bc |
| Half local conc. | 14.46c | 14.24c | 14.36c | 13.68c | 14.28c | 14.62c |
| Twice local conc. | 38.23a | 39.92a | 38.78a | 24.68a | 27.39a | 29.22a |

In columns, different letters indicate significant difference at 5% level.

Table 12: Soil potassium (cmol/kg) at 3 Months and 12 Months after dye application

| Treatment | At 3 months | | | At 12 Months | | |
|-------------------|-------------|---------|---------|--------------|---------|---------|
| | 0-10cm | 10-20cm | 20-30cm | 0-10cm | 10-20cm | 20-30cm |
| Control | 0.42b | 0.37c | 0.33b | 0.41b | 0.39c | 0.37b |
| Local conc. | 35.17a | 24.00b | 26.00ab | 24.00a | 23.33ab | 30.67a |
| Spent local conc. | 35.17a | 29.67ab | 31.33a | 32.67a | 32.00a | 41.27a |
| Half local conc. | 49.67a | 34.60a | 34.80a | 39.00a | 27.33ab | 29.27a |
| Twice local conc. | 31.37a | 24.53b | 42.03a | 24.00a | 20.00b | 25.93a |

In columns, different letters indicate significant difference at 5% level.

Table 13: Soil magnesium (cmol/kg) at 3 Months and 12 Months after dye application

| Treatment | At 3 months | | | At 12 Months | | |
|-------------------|-------------|---------|---------|--------------|---------|---------|
| | 0-10cm | 10-20cm | 20-30cm | 0-10cm | 10-20cm | 20-30cm |
| Control | 0.40c | 0.36c | 0.36c | 0.38c | 0.36c | 0.45c |
| Local conc. | 90.59b | 93.78b | 96.80b | 94.26b | 95.59b | 93.17b |
| Spent local conc. | 99.50a | 98.52a | 98.40a | 100.63a | 98.62b | 98.34a |
| Half local conc. | 95.62ab | 98.07ab | 97.91ab | 100.60a | 99.60a | 98.25a |
| Twice local conc. | 92.17ab | 96.77ab | 97.21ab | 95.99b | 93.90b | 92.65b |

In columns, different letters indicate significant difference at 5% level.

Table 14: Sulphur (mg/kg) at 3 Months and 12 Months after dye application

| Treatment | At 3 months | | | At 12 Months | | |
|-------------------|-------------|---------|---------|--------------|---------|---------|
| | 0-10cm | 10-20cm | 20-30cm | 0-10cm | 10-20cm | 20-30cm |
| Control | 0.74d | 0.78c | 0.81c | 0.88c | 0.93d | 0.91c |
| Local conc. | 6.64b | 8.13a | 8.06a | 7.56a | 7.44ab | 7.68a |
| Spent local conc. | 6.73b | 7.42ab | 7.98a | 7.01a | 6.86b | 7.21ab |
| Half local conc. | 6.05c | 6.16b | 6.06b | 4.61b | 4.48c | 6.55b |
| Twice local conc. | 8.93a | 8.20a | 8.15a | 7.47a | 7.90a | 8.01a |

In columns, different letters indicate significant difference at 5% level.

Table 15: Soil pH at 3 Months and 12 Months after dye application

| Treatment | At 3 months | | | At 12 Months | | |
|-------------------|-------------|---------|---------|--------------|----------|---------|
| | 0-10cm | 10-20cm | 20-30cm | 0-10cm | 10-20cm | 20-30cm |
| Control | 7.00a | 7.00a | 7.00a | 6.97ab | 7.00a | 7.00a |
| Local conc. | 6.37c | 6.30b | 6.23b | 7.13a | 7.03a | 6.90ab |
| Spent local conc. | 6.33c | 6.33b | 6.37b | 6.77b | 6.83ab | 6.93ab |
| Half local conc. | 6.43bc | 6.33b | 3.23b | 6.77b | 6.80aabb | 6.87bc |
| Twice local conc. | 6.53b | 6.23b | 6.13b | 6.37c | 6.53b | 6.77c |

In columns, different letters indicate significant difference at 5% level.

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