Chemical Characterization of Soil Used as a Deposit for Waste Originating From the Manufacturing of Products Based on Fiberglass

Companies need to follow environmental laws as they affect their manufacturing systems, waste handling, and the management of soil on their properties to ensure that their properties are either improved or altered as little as possible. Thus, the objective of this study was to evaluate the use of energy dispersive X-ray fluorescence spectrometry (EDXRF) to determine the concentrations of elements present in the soil at a manufacturer of products based on fiberglass where wastes from production activities have been deposited on the property.

Use of Energy Dispersive X-Ray Fluorescence Spectrometry Proves Effective for Contaminant Monitoring

Introduction

The effects of inappropriate landfilling of waste on soil and in surface waters are recognized as among the most serious problems of our time. The effects of the release of waste into nature are incalculable and are reflected primarily by

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contamination of the water and soil and, in turn, on the increasing prevalence of diseases associated with environmental pollution (Oliveira & Jucá, 2004).

The presence of metals in the soil occurs naturally from the weathering of an area's original rocks. However, elevated metals contents have frequently been observed in some areas as a result of anthropogenic activities, primarily involving the deposition of industrial wastes directly onto the ground (Reis et al., 2007).

The proper disposal of wastes is necessary to avoid environmental pollution and contamination, and predictions must be made of the behavior of heavy metals that have been deposited on the soil.

There is the possibility that heavy metals can leach into the soils where they were applied or deposited (Bertoncini & Mattiazzo, 1999). Thus, the proper disposal of wastes is necessary to avoid environmental pollution and contamination, and predictions must be made of the behavior of heavy metals that have been deposited on the soil. The redistribution of the metals within the soil as they transform from unstable forms to more stable forms depends on the metal species, the soil properties (e.g., pH, cation-exchange capacity, and organic matter), and the duration of exposure (McBride, 1995).

Metals Used in the Manufacture of Fiberglass-Based Products

According to Araúja, Araúja, Pereira, Ribeiro, and deMelo (2006), the increasing production of consumer goods generates a large volume of waste from the manufacturing and sale of goods. While the recycling of materials is an alternative to the reuse of these "wastes," and consequently plays a role in reducing the environmental impact that such wastes can cause, Araújo et al.

(2006) claim that just 50% of such wastes arrive at sanitary landfills. Furthermore, a large quantity of industrial wastes include plastics, which are mixed with other materials and additives to form composites. In this condition, recycling of the component materials becomes more difficult (Forlin & Faria, 2002).

In this context, fiberglass, a plastic matrix reinforced with glass fibers, which is made by the agglomeration of extremely thin glass filaments added to polyester resin or other types of resins, has a chemical composition consisting primarily of 52–56% silicon dioxide or silica (SiO_2), 12–16% aluminum oxide (Al_2O_3), 16–25% calcium oxide (CaO), and 8–13% boron trioxide (B_2O_3) (Orth, Baldin, & Zanotelli, 2014; Smith, 2000).

In the process of manufacturing fiberglass-based products, the glass fibers used to reinforce the product receive a covering called "encimagem," which improves resistance and is made with coupling agents compatible with the polyester resins, vinyl ester, and epoxy. The short glass fiber type E (E-glass), obtained from a mixture of oxides of silicon (Si), aluminum (Al), boron (B), calcium (Ca), and magnesium (Mg) (e.g., borosilicate of alumina and calcium), is usually used as thermoplastic reinforcements as low-cost alternatives to aramid and carbon (Larena, De la Ordem, & Urreaga, 1992), and it results in improved material properties, such as impact resistance and rigidity (Pizzitola, Machado, & Wiebeck, 2011; Wambua, Ivens, & Verpoest, 2003).

Other commercialized glass fibers were analyzed by Dovlitova, Boldyreva, and Malakhov (2011). One of these had in its composition sodium and aluminum silicates in concentrations of 75–77% SiO_2, 17–19% sodium oxide (Na_2O), 3–5% Al_2O_3, and <1% CaO. Another had as its composition

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sodium zirconium silicate and titanium with concentrations of 66–70% SiO_2, 12–24% Na_2O, and 9–14% zirconium dioxide (ZrO_2). In addition to these main components, the commercial glass fibers showed impurities in the form of oxides: Ca, Mg, iron (Fe), and titanium (Ti), with total concentrations of 1.0–2.0%.

Analytical Techniques for Characterizing Glass Fiber

Many analytical techniques may be used to determine the chemical composition and structure of glass fiber, including infrared spectroscopy, X-ray photoelectron spectroscopy, nuclear magnetic resonance spectroscopy, and atomic emission spectroscopy by inductively coupled plasma (Glazneva et al., 2012).

The conventional techniques used for determining the chemical composition are time consuming, result in high costs for reagents, and require several steps to quantify all of the elements. EDXRF stands out as an important alternative method to streamline the process of chemical characterization of fiberglass waste by allowing for more rapid analysis than other methods. Increasingly, it is being used in the chemical characterization of various materials, such as soil, rocks (Dantas, Dantas, Van't Dack, & Van Griekem, 1981; Wastowski, da Rosa, Cherubin, & Rigon, 2010), ceramics, biological materials (Albers, Melchiades, Machado, Baldo, & Boschi, 2002; Ferreira, Fabris, Santana, & Curi, 2003), particles suspended in the air (Adeloju, Bond, & Briggs, 1985; Bona, Sarkis, & Salvador, 2007), and liquids (Pataca, Bortoleto, & Bueno, 2005). This new EDXRF technique offers several advantages in the chemical analysis of elements, such as:

- Adaptability to automation,

- Multielement quick analysis (very important because of the interdependence among the micronutrients in biological systems),
- Simplified sample preparation,
- Detectability limits within those required for many biological samples, and
- Low reagent and time costs.

For analyses of glass fiber composition, Luo et al. (2011) successfully used a portable X-ray fluorescence spectrometer by dispersive energy to identify the elements Na and Mg that were used in the characterization of 58 glass vase fragments found in Xinjiang, China. Mashin, Onicheva, Tumanova, and Rudnevskii (2000) conducted a procedure for determining chalcogenide glass composition using X-ray fluorescence spectrometry. The developed methodology was used to monitor the technological conditions for the production of chalcogenide glasses and subsequent monitoring of the composition in the preparation process; this control is performed with high speed, without destruction of the sample, and with the necessary precision.

EDXRF stands out as an important alternative method to streamline the process of chemical characterization of fiberglass waste by allowing for more rapid analysis than other methods.

The objective of this study was to use EDXRF to identify the possible accumulation of chemical elements in the soil due to the deposition of residues of fiberglass-based products by industry.

Study Location

This study was conducted at the Bakof Industry and Trade of Fiberglass Ltda in the city of Frederico Westphalen, in the Brazilian state of Rio Grande do Sul (RS). The company is involved in the manufacturing of water tanks, dish antennas, septic tanks,

tanks for washing, among other items. These products are manufactured using chemical components derived from polyethylene and fiberglass. The study area is located at coordinates 27° 23'06.86 South, 53° 23'45.08 West, at an altitude of 658 meters (m) above mean sea level, in the city of Frederico Westphalen.

Materials and Methods

Sample Collection

Samples of soil and deposited waste were collected from the studied area to assess the concentrations of chemicals in the soil. Collection of soil samples was performed using an auger at 11 sampling points (P1–P11) that best represent the area under study as shown in **Exhibit 1**. These sampling points were georeferenced with a GARMIN global positioning satellite device.

Samples of soil and deposited waste were collected in the studied area to assess the concentrations of chemicals in the soil.

At every point, two samples were collected at depths of 0–15 centimeters (cm) and 15–30 cm. Each sample was placed in a plastic bag and marked with its location and depth. At points 5 and 9, the samples were collected at depths of 0–2 cm, and at point 7, at a depth of 0–10 cm. These variations in sampling depth were required due to the presence of an outcropping of matrix rock of basaltic formation (the C horizon), which made it impossible to collect soil samples at greater depths in these locations.

"Witness" and Offsite Soil Sample Collection

Sampling point P12 was chosen to function as a "witness" or control sample because its location is some distance away from the waste disposal area on the company's property. According to the company's historical records of soil use, that particular area has not been influenced by an-

thropogenic activities, such as the disposal of wastes or scraps from its manufacturing processes. In addition, soil from a native forest, which was collected 2 kilometers distant from the study area (sample ST), was used to confirm the presence of chemical elements in the soil of the region, and thus provide a comparison to the observed results from analyses of the samples collected in the study area.
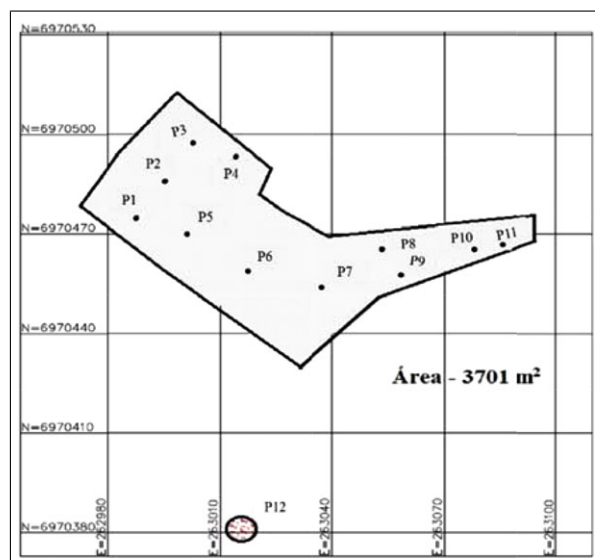
Waste Sample Collection

Samples of wastes from the company's manufacturing processes, which are being deposited on the ground in the study area, were also collected for analyses. These wastes were defined as basic blue residue (RBA) and fiber waste (RF), and its physical composition is formed by the agglomeration of very thin glass filaments added to polyester resin or other resins. These wastes were analyzed, and the results of their chemical compositions were used for statistical evaluation with the values observed in the soil samples by using Pearson's correlation coefficient.

Sample Analyses

Analyses were performed in the Research and Chemical Analysis Laboratory (LAPAQ), located on the premises of the Center for Higher Education North (CESNORS) of Federal University of Santa Maria (UFSM). Soil chemical composition was evaluated with an emphasis on those chemical elements that are detrimental to organic elements. Before analysis, the samples were oven-dried at 105 degrees Celsius (°C) for 24 hours until they achieved constant weights. Posteriorly, they were ground and pressed (10 tons for 10 minutes) to form tablets. These tablets were used for chemical analysis in the EDX-720 equipment of the EDXRF (Wastowski et al., 2010).

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Exhibit 1. Georeferenced Sketching of the Study Site and the Sample Collection Points

Results and Discussion

The table in **Exhibit 2** shows the chemical element concentrations present in the soil profile in the layers from 0–15 cm to 15–30 cm along with the chemicals found in the industrial wastes (RBA and RF) collected at the site.

The studied soil is classified as red latosol, and it has in its structural composition high concentrations of iron oxide and clay (Streck et al., 2008), which explains the high levels of iron and silicon found in the analyses of soils collected from all sampling points. As **Exhibit 3** shows, at the points where the iron and silicon contents are significant, we can observe the declivity of the terrain, where an increased flow of rainfall water and consequent surface runoff has resulted in decreased soil organic matter. The quantification of the flows and their directions reveal the composition and origin of the sediments. A key challenge in this context is the quantification of human impacts caused in the area (Brown et al., 2009).

The table in Exhibit 2 shows a great presence of silicon and calcium in the composition of the RBA and RF, but these elements have not contributed significantly to the alteration of the elements in the soil. This statement was confirmed by the analysis of the correlation between the elements present in the soil of the study area with the elements present in the nonanthropic soil, a correlation between the factors of 0.97.

The chemicals commonly associated with toxicity or soil pollution are arsenic (Ar), cadmium (Cd), cobalt (Co), chromium (Cr), copper (Cu), lead (Pb), mercury (Hg), molybdenum (Mo), nickel (Ni), selenium (Se), and zinc (Zn) (Nellessen & Fletcher, 1993). The table in Exhibit 2 shows that Ar, Cd, Co, Pb, Hg, Mo, Ni, and Se were not found in the soil analyzed for this study. In fact, from the elements cited by Nellessen and Fletcher (1993), only Cr, Cu, and Zn were found. Of these, only Cu and Zn are present in the composition of RBA and RF (Nellessen & Fletcher, 1993). Cr is not detected in the composition of the latter two

Exhibit 2. Results of Soil Analyses of the Soil Profiles Collected at Depths of 0–15 cm and 15–30 cm Determined by EDXRF

| Depth 0–15 cm milligrams per kilogram (mg/kg)														
	RBA	RF	P1	P2	P3	P4	P5	P6	P7	P8	P9	P10	P11	P12	ST
Si	53.80	55.66	157.08	156.02	152.01	123.49	150.92	167.86	148.06	144.01	119.12	169.50	178.5	171.66	176.14
Fe	2.25	1.13	147.53	143.51	158.70	112.79	112.87	128.33	142.58	163.24	119.56	189.06	182.4	121.98	152.94
Al	1.,90	13.14	99.95	110.74	110.07	80.51	82.44	102.40	84.73	108.98	76.21	89.88	88.46	88.26	64.75
Ti	3.33	0.04	16.96	13.04	14.79	13.32	12.09	9.91	14.44	19.59	13.93	24.74	25.97	10.79	37.02
Ca	77.63	72.31	5.65	6.84	7.07	3.61	16.34	7.60	3.53	2.77	4.95	2.79	1.68	5.71	2.82
Ba	nd	nd	4.50	7.77	8.72	4.05	5.50	2.81	6.40	4.85	7.61	11.08	11.68	5.02	13.22
S	2.48	2.42	3.71	3.28	5.64	2.76	2.34	4.40	5.03	3.56	3.07	7.51	3.93	4.22	6.37
Mn	0.04	0.03	2.37	2.30	3.11	2.15	2.04	2.02	3.00	2.98	2.61	4.17	4.28	2.98	4.33
K	0.60	0.71	1.55	2.15	1.42	1.20	2.08	5.26	2.87	1.10	1.83	2.41	2.70	8.59	1.57
V	nd	nd	0.97	1.03	1.07	0.78	0.77	0.63	0.90	1.07	0.98	1.52	1.50	0.69	1.81
Cu	0.06	0.05	0.51	0.44	nd	0.36	nd	0.45	0.40	0.47	0.32	0.50	0.45	0.41	0.43
Zr	0.03	nd	0.40	0.41	0.44	0.32	0.35	0.39	0.42	0.51	0.38	0.64	0.66	0.38	0.71
Zn	0.03	0.04	0.21	0.20	0.21	0.15	nd	0.19	0.16	0.21	0.15	0.29	0.20	0.16	0.22
Cr	nd	nd	0.12	0.12	0.14	0.11	0.09	0.15	0.12	0.14	0.09	0.09	0.10	0.10	0.14
Sr	0.16	0.36	0.09	0.09	0.09	0.07	0.16	0.08	0.04	nd	0.06	nd	nd	0.07	0.04
Depth 15–30 cm milligrams per kilogram (mg/kg)														
Si	53.80	55.66	164.69	157.08	152.06	141.88		159.87		149.30		169.30	165.53	164.69	176.14
Fe	2.25	1.13	115.36	138.42	147.44	127.84		132.86		172.93		167.24	180.38	115.36	152.94
Al	10.90	13.14	85.90	111.99	106.74	91.14		92.76		110.22		87.69	84.02	85.90	64.75
Ti	3.33	0.04	8.40	13.47	15.21	14.10		12.87		19.08		22.76	26.61	8.40	37.02
Ca	77.63	72.31	5.44	6.87	4.25	4.74		9.81		2.82		2.57	1.62	5.44	2.82
Ba	nd	nd	5.11	6.50	6.92	5.81		5.78		7.81		7.61	9.16	5.11	13.22
S	2,48	2.42	3.50	4.00	3.78	3.18		6.84		5.26		4.89	3.74	3.50	6.37
Mn	0.04	0.03	2.55	2.25	2.69	2.85		2.70		3.22		3.61	4.47	2.57	4.33
K	0.60	0.71	8.46	2.16	1.71	1.52		4.50		1.26		2.83	2.34	8.46	1.57
V	nd	nd	0.74	0.89	0.94	0.79		7.98		1.10		1.43	1.42	0.74	1.81
Cu	0.06	0.05	nd	0.42	0.47	0.42		0.47		0.51		0.42	0.48	nd	0.43
Zr	0.03	nd	0.37	0.42	0.41	0.35		0.38		0.53		0.55	0.64	0.37	0.71
Zn	0.03	0.04	0.14	0.20	0.20	0.16		0.30		0.20		0.19	0.22	0.14	0.22
Cr	nd	nd	0.11	0.14	0.15	0.15		0.12		0.14		0.09	0.11	0.11	0.14
Sr	0.16	0.36	0.05	0.08	0.08	0.12		0.09		nd		nd	nd	0.05	0.0

Abbreviations: RBA, blue base residue; F, fiber residue; ST, witness soil; nd, not detected.
*Values of points 5, 7, and 9 in the depth of 15–30 do not exist because of rock outcrop (C horizon).

wastes. However, the levels of Cu and Zn found in the soil samples collected from the sampling area do not differ statistically from the values observed in the soil collected from the witness or control soil sample, P12, or in the soil collected in the native forest area, ST, meaning that Cr, Cu, and Zn are part of the natural composition of the soil.

The chemical element Al showed increases in concentrations in the soil collected from points 6 to 8 when compared to the soil collected from the native forest site, ST; however, Al was not present in the samples in concentrations that would characterize it as a soil contaminant. The increase in aluminum levels can be related to the reduction of organic matter in that portion of the study area, as the soil area is uncovered (without vegetation coverage) and subject to the action of runoff. Such a result was also observed by Lima et al. (2007), where the authors claimed that with the reduction the

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Exhibit 3. Tracks of Concentrations of Iron and Silica Over the Area in the Direction of Land Declivity
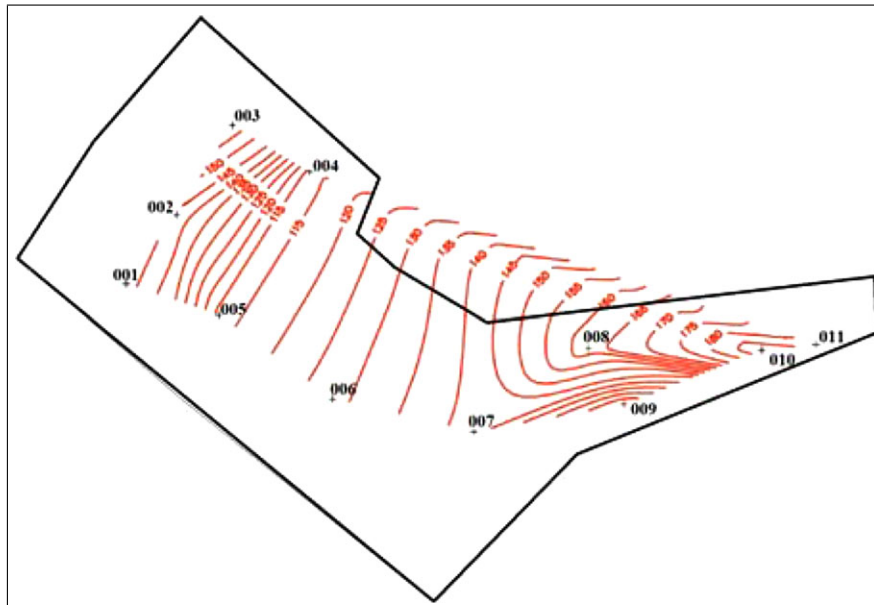
organic matter, the aluminum content in the analyzed soil increased.

According to Sharpley et al. (2008), losses of nutrients in the soil happen because the nutrients are connected to the soil particles, thereby reaffirming that erosion control measures should be prioritized, both to avoid the loss of nutrients and to avoid contamination of disposal areas.

Correlation Analysis

A correlation analysis between the profiles of soil collected from 0 to 15 cm deep and from 15 to 30 cm deep revealed differences between the depth profiles. A change in soil texture, which is characterized as pedogenic evolution (Kalbus et al., 2012), is apparent. In contrast, there was no leaching of chemicals from the surface horizon to the subsurface horizon, thereby demonstrating that the elements present in the composition of the RBA and RF wastes did not contribute to soil contamination.

The data from the statistical correlation analysis, which is shown in the table in **Exhibit 4**, allow us to conclude

that contamination by elements from the products used at the study location is not present in the soil. In the soil samples collected at the two analyzed depths (i.e., 0–15 cm and 15–30 cm), the observed correlation between the soil collected in the study area and the soil from the witness and control samples (P12 and ST) was on average 0.90, and the correlation between these soils and the waste products analyzed (i.e., RBA and RF) was on average 0.30 for the two sampling depths. These results differ from what we expected regarding the classification of waste, a II A-class of pollutants, which is not inert, and that, over time, undergoes some sort of change or decomposition (Zilber, Caruzzo, & De Abreu Campanário, 2011).

We can infer that these results are due to the fact that the wastes deposited and in contact with the soil in the study area were not deposited in a particle size that would allow for its immediate weathering, or that the exposure time of these products to the soil (less than 15 years) has been insufficient to have allowed the weathering of elements

Exhibit 4. Correlation Between Chemical Elements Present in the Sample Profiles at Depths 0–15 cm and 15–30 cm in the Soil With Wastes (RBA and RF)

Depth 0–15 cm milligrams per kilogram (mg/kg)													
	RBA	RF	P1	P2	P3	P4	P5	P6	P7	P8	P9	P10	P11	P12
Soil	0.298	0.309	0.977	0.965	0.965	0.975	0.974	0.968	0.984	0.962	0.978	0.985	0.991	0.976
RBA	1.000	0.997	0.285	0.289	0.240	0.285	0.349	0.335	0.276	0.202	0.273	0.216	0.236	0.348
RF	–	1.000	0.302	0.308	0.250	0.303	0.362	0.359	0.292	0.210	0.287	0.221	0.243	0.372
P1	–	–	1.000	0.998	0.998	1	0.991	0.994	0.999	0.995	0.999	0.989	0.991	0.988
P2	–	–	–	1.000	0.998	0.999	0.990	0.995	0.994	0.994	0.997	0.980	0.983	0.986
P3	–	–	–	–	1.000	0.997	0.982	0.987	0.996	0.998	0.999	0.988	0.987	0.977
P4	–			–	–	1.000	0.991	0.995	0.997	0.994	0.998	0.986	0.989	0.989
P5	–	–	–	–	–	–	1.000	0.997	0.988	0.971	0.986	0.968	0.977	0.997
P6	–	–	–	–	–	–	–	1.000	0.990	0.979	0.989	0.970	0.976	0.997
P7	–	–	–	–	–	–	–	–	1.000	0.993	0.999	0.995	0.996	0.987
P8	–	–	–	–	–	–	–		–	1.000	0.997	0.989	0.987	0.967
P9	–	–	–	–	–	–	–	–	–	–	1.000	0.993	0.994	0.983
P10	–	–	–	–	–	–	–	–	–	–	–	1.000	0.999	0.968
P11	–	–	–	–	–	–	–	–	–	–	–	–	1.000	0.977
P12	–	–	–	–	–	–	–	–	–	–	–	–	–	1.000
Depth 15–30 cm milligrams per kilogram (mg/kg)													
Soil	0.298	0.309	0.973	0.963	0.969	0.975	*	0.978	*	0.964	*	0.990	0.988	0.973
RBA	1.000	0.997	0.331	0.296	0.268	0.292	*	0.331	*	0.199	*	0.246	0.219	0.331
RF	–	1.000	0.353	0.317	0.285	0.310	*	0.351	*	0.205	*	0.255	0.223	0.353
P1	–	–	1.000	0.997	0.999	1.000	*	0.997	*	0.994	*	0.995	0.989	0.987
P2	–	–	–	1.000	0.999	0.999	*	0.995	*	0.992	*	0.988	0.979	0.986
P3	–	–	–	–	1.000	0.997	*	0.991	*	0.998	*	0.991	0.986	0.976
P4	–	–	–	–	–	1.000	*	0.997	*	0.993	*	0.993	0.986	0.988
P5	–	–	–	–	–	–	*							
P6	–	–	–	–	–	–	–	1.000	–	0.970	–	0.983	0.969	0.997
P7	–	–	–	–	–	–	–	–	*					
P8	–	–	–	–	–	–	–	–	–	1.000	*	0.983	0.970	0.997
P9	–	–	–	–	–	–	–	–	–	–	*			
P10	–	–	–	–	–	–	–	–	–	–	–	1.000	0.994	0.986
P11	–	–	–	–	–	–	–	–	–	–	–	–	1.000	0.965
P12	–	–	–	–	–	–	–	–	–	–	–	–	–	1.000

Abbreviations: RBA, blue base residue; F, fiber residue; ST, witness soil; nd, not detected.
*Values of points 5, 7, and 9 in the depth of 15–30 do not exist because of rock outcrop (C horizon).

from the RBA and RF wastes and their subsequent leaching into the soil.

Conclusions

The chemical elements in the wastes of the blue base and fiber residues that make up fiberglass products did not contaminate the soil where they were deposited. One can infer that the absence of correlation between the chemical elements of the industrial waste and the soil may be related to the short exposure time at the study site.

The variations in the concentrations of the chemical elements among the sampling points in the study area (points P1 to P11) are related to weathering (runoff of rainfall water) caused by anthropogenic influence on site and not to the leaching of the chemical elements from the wastes deposited there.

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Finally, use of the EDXRF is efficient for monitoring areas with possible environmental contamination by chemicals.

Acknowledgments

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