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Investigation of different amendments for dump reclamation in Northern Vietnam

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ABSTRACT

Giant spoil dumps originate in the course of open-cast mining. The initial properties of Triassic spoil as well as pedogenesis are characterized by weathering processes. Due to the poor conditions in dump spoil a natural succession of plants or a re-vegetation is a lengthy process. The Chinh Bac field experiment (Ha Long City, Quang Ninh province, Vietnam) was planned to investigate the possible impacts of three amendments – charred rice straw, power station ashes and fine material originating from the mining area in combination with sieved spoil of substrate quality – to enhance plant growth. The main focus of this study was directed towards the impact of added amendments on spoil chemical parameters. The investigations demonstrated that simply sieving the spoil leads to better substrate conditions. It increases the fine material which in turn leads to easier plantation conditions. At the same time oxidative processes and leaching acid products are stimulated which raises the pH values afterwards. The application of charred rice straw increased the amount of alkaline cations, in particular potassium, boosting the pH value. This led to an enhanced supply of nutrients for the plants in comparison to the other amendment variations which were very poor in nutrient availability. Adding power station ash resulted in a short-term pH value increase, however potential pollution caused by heavy metals cannot be ruled out. The pyrite containing fine material was identified as the poorest amendment. It may allow vegetation to grow quickly, however the proceeding weathering processes cause a strong acidification. This mobilizes heavy metal and aluminum ions which prevent healthy plant growth.

1. Introduction

The Quang Ninh province (Fig. 1) – longitude 106°25′ to 108°25′ east and latitude 20°40′ to 21°40′ north – in the north-east of Vietnam is famous for Ha Long Bay, which has been a UNESCO world natural heritage site since 1994. Thus the nature of this region must be protected. On the other hand the region's main industrial sector is coal mining. Coal is removed in large parts in open-cast mining causing drastic disturbances of entire landscapes. Sulphurous minerals accompanying the coal are mixed with the other spoil and are deposited during the dumping process. The spoil mainly consists of coaly and pyritic material. In consequence, the top-layer material in vast areas contains large amounts of ferrous sulphides (pyrite) that oxidize. This weathering process leads to a mobilization of free mineral acids as well as high quantities of sulphate and iron (Evangelau, 1995; Häge, 1996; Hüttl, 1998). Hence, it creates poor ecological site conditions characterized by very low pH values of around 3 after exposing pyrite to water and oxygen as well as due to the high electrical conductivity (EC), low nutrient availability (especially N and P) and the enhanced solubility of heavy metals and aluminum (Deka Boruah, 2006; Fitter, 1974; Gogoi et al., 2007; Jha and Sing, 1991; Kent, 1982; Palmer, 1976; Raju and Hassan, 2003; Sheoran et al., 2010; Williams and Cooper, 1976).

As a result of a high skeletal content and a lack of fine material such as clay, silt and sand the mining waste material, in most cases, has a relatively low water-holding capacity (Dept. of Environment et al., 1996; Moffat and McNeill, 1994). Over time the spoil on the dump plateau is highly compacted. Otherwise the steeply-piled dumps are vulnerable to erosion and complicate restoration. Heavy rainfall can cause matter losses on slopes due to surface water run-off (Sheoran et al., 2008, 2010; Wong, 2003). Furthermore, dust development and sedimentation impair the water and aerial quality leading to rising pressure to restore regions no longer used by the coal industry.

Ecological succession in open-cast coal mines is a lengthy process (Dowarah et al., 2009; Jha and Singh, 1992; Wali, 1979). The aims of restoration are to create stable and sustainable ecosystems (Haigh, 1993) and to establish stable nutrient cycles from plant growth and microbial processes (Kavamura and Esposito, 2010; Lone et al., 2008; Singh et al., 2002).

Re-vegetation reduces air and water pollution, stabilizes surfaces and rapidly improves the appearance of the sites (Kent, 1982; Wong, 2003). On the other hand Tropek et al. (2010) and Doležalová et al. (2012) favor a natural succession of open-cast mining areas in terms of advanced biodiversity. They found a higher species richness in comparison to technically restored dumps in the Czech Republic.

In the case of a fast restoration an improvement of spoil characteristics is helpful for a successful re-vegetation. Reclamation strategies must address soil structure, soil chemistry and soil fertility to restore the land as closely as possible to its pre-mining condition and for it to

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Fig. 1. Location of study area — Vietnam Quang Ninh province (http://en.wikipedia.org/wiki/Quang_Ninh_province).

develop into a sustainable ecosystem (Sheoran et al., 2010). The spoil productivity can be improved by adding various amendments.

Melioration with high doses of alkaline materials leads to a higher pH value (Illner and Lorenz, 1965; Schaaf et al., 1999). Power station ash (PSA) acts as an alternative to lime for reclaiming the acidic mine spoils (Carlson and Adriano, 1993; Haering and Daniels, 1991; Stehouwer et al., 1995a, 1995b). High amounts – approximately 550 Mt a^{-1} – of alkaline PSA are produced by coal combustion worldwide (Querol et al., 2001). PSA itself is a problematic solid waste material because it contains contaminants (i.e. heavy metals). Conversely many studies recommend PSA as a suitable amendment to reclaim wastelands and dumps with problematic conditions for plant growth (Brown et al., 1997; Carlson and Adriano, 1993; Fail and Wochok, 1977; Goyal et al., 2002; Gupta et al., 2000; Jala and Goyal, 2006; Jastrow et al., 1981; Ram and Masto, 2010; Ram et al., 2006; Saxena et al., 1997). The overall beneficial effects of PSA are primarily associated with improved physicochemical and biological characteristics of the mine spoil (Ram and Masto, 2010).

In a study of Maiti and Saxena (1998) the suitability of domestic raw sewage and a grass-legume mixture as an alternative to topsoil was investigated. The quality of the mine spoil improved concerning a higher pH value, the accumulation of organic carbon and therefore a higher field moisture capacity. Otherwise more heavy metals accumulated in the top spoil horizon. Juwarkar and Jambuhulkar (2008) postulated that applying 50 t/ha of effluent treatment plant sludge improved the physicochemical properties of the coal mine spoil and reduced the toxicity of heavy metals thanks to a higher organic matter content and pH value.

The potential of biochars for restoring contaminated soils was investigated by Beesley et al. (2011). Due to its high specific surface area biochar serves as an absorbent of contaminants and contributes to reducing pollutant mobility. Despite the increased retention of plant nutrients, additional fertilizers such as compost, manure or other amendments are required. The effect of biochar on soil properties has been investigated in many studies (Asai et. al., 2009; Glaser et al., 2002; Lehmann et al., 2003; Steiner et al., 2007; Warnock et al., 2007). In a study by Masulili et al. (2012) rice husk biochar was investigated in a laboratory experiment as a soil amendment to raise the pH value, to improve the water-holding capacity and to foster soil organisms. The results of a laboratory experiment show a significant improvement of spoil properties respectively reducing bulk density, soil strength, exchangeable Al, soil organic matter and more.

This study focuses on the investigation of three different locally-available amendments in comparison to pure spoil material that was only sieved in combination with three plant species for improving the chemical and physical properties of spoil to cost-efficiently restore and re-vegetate ready-built spoil areas. The economic optimum of these substrate amendments to increase the pH value was determined during two pre-experiments under laboratory conditions and finally tested in a field experiment. The investigated amendments were a) the sieved pure spoil material (W), b) charred rice straw (CRS), c) power station ash (PSA) and d) fine spoil material (FSM) from a different location in the open-cast mining area. For treatments b) to d) the material was mixed with sieved spoil material from the experiment site.

It was assumed that only sieving the spoil leads to improved spoil conditions with regard to lower bulk density, a higher amount of fine material and enhanced water-holding capacity. The input of amendments into treatments with CRS and PSA were expected to significantly improve the spoil conditions in comparison to treatment W. The commonly used PSA amendment is a simple method to rapidly increase pH values and reduce the solubility of pollutants, but its potential as an amendment for restoring Vietnamese dumps is yet to be proven. The CRS treatment was assumed to have a similar effect on spoil conditions to that of the biochar amendment on contaminated soils presented by Beesley et al. (2011) and to that of charred rice husk presented by Masulili et al. (2012). The supposedly increased specific surface area of CRS should retain pollutions and simultaneously provide plant nutrients, especially potassium, which is contained in rice straw in high concentrations. CRS is often used by farmers but usually unknown by managers who are in authority for recultivation.

The present restoration approach of Vietnamese engineers uses unconsolidated FSM from other sites in the open-cast mining area. The advantage of FSM is a fast plantation. However the assumed disadvantage is the continuous weathering processes of FSM which lead to more acidity in the spoil and poor substrate conditions.

2. Materials and methods

The substrate used for the two pre-experiments was taken from the Chinh Bac dump belonging to the Nui Beo coal company in the VINACOMIN group in Ha Long (Vietnam). The mine spoil (Triassic — sub formation *T3n-r hg1*) was deposited around 5 years ago and its pyrite has undergone oxidation since then causing an increased acidity of the spoil and high amounts of dissolved heavy metals. A substrate sample (mixture of approximately 10 subsamples) was collected in November 2008 from the same area where the field experiment was to be installed in March 2009. The sample (approximately

Table 1
Results and standard deviation(s) of estimated biological parameters microbial biomass (C_{mic}), dry matter (DM) of above-ground biomass and roots of climatic chamber experiment (n = 3).

	Method	Unit	0.015 M% PSA	0.06 M% PSA	0.1 M% CRS	0.3 M% CRS
C_{mic}	Average	μg C	19.41	23.66	20.93	22.79
	s	g DM^{-1}	1.85	3.37	2.65	2.06
DM above-ground biomass	Average	g	0.023	0.089	0.051	0.197
	s		0.027	0.031	0.065	0.047
DM roots	Average	g	0.026	0.094	0.047	0.186
	s		0.016	0.021	0.071	0.050

10 kg from 0 to 20 cm depth) was air-dried and sieved smaller than 7 mm. An X-ray fluorescence spectroscopy (XRF) analysis was carried out to roughly estimate the total element concentrations (S, Pb, Ni, As and CaO) with WDXRF: S4 Pioneer, Germany and EDXRF: XLAB 2000 Germany in accordance with DIN EN 15309.

2.1. Pre-experiments

2.1.1. pH test series

In the first pre-experiment the pH values of substrate mixtures with dump substrate and different amounts of added PSA and CRS were measured. The result of this pre-experiment served to quantify the amount of amendments necessary to neutralize the substrate for the treatment with PSA and to achieve a visible rise in pH for the CRS treatment. The pH value of these substrate mixtures was determined potentiometrically (METTLER TOLEDO, Seven Easy, Germany) in a suspension with 0.01 m $CaCl_2$ at an SSR of 1:2.5 (DIN 19684).

2.1.2. Climatic chamber experiment

In a second pre-experiment from February to April 2009 the effect of the two amendments, PSA and CRS, on soil parameters and plant growth of *Lolium perenne* as a common test plant was investigated in a climatic chamber. The original substrate from the dump was mixed with a high (0.060 M% PSA and 0.3 M% CRS respectively) and a low amount (0.015 M% PSA and 0.1 M% CRS respectively) of each amendment. The high level of amendment corresponds with the amount of PSA necessary to neutralize the dump substrate and the high amount of CRS leads to a surge in the pH value considering the operational availability of this amendment respectively. The low amendment level was included for economic reasons to test whether a lower amount of amendment would suffice for vigorous plant growth. Approximately 3500 g of substrate mixture were filled in a 2-liter compartment and equipped with suction cups. The bulk density of 1.75 g/cm^3 corresponds to that of the dump site. Each treatment was repeated in three compartments. Due to the limitation on 12 places no implementation of a control variant was possible. 0.5 g seeds of *L. perenne* were sown after two days. All compartments stood on a balance to ensure a constant water supply for the plants. Due to poor germination and plant growth all compartments were fertilized after 4 weeks with 50 mg N/kg soil and after 5 weeks with 34 mg N/kg soil. The soil solutions were sampled weekly with micro-suction cups to measure pH, chloride, nitrate, ammonium, phosphate, and the elements As, Ca, Fe, K, Na, Mg, Mn, and S of each soil substrate mixture treatment out of the 3 compartments. It was necessary to take mixture samples of the three compartments of one soil mixture treatment because of the low amount of soil solutions sampled weekly. The pH value was estimated for each soil compartment individually.

The pH values of the sampled soil eluates were measured with a pH-meter (IQ Scientific Instruments, IQ 240 pH-meter, USA) (Fig. 3). The soluble anions chloride and nitrate were analyzed with an ion chromatograph DX 500 (DIONEX, USA) according to the DIN EN ISO 10304-2 protocol. Ammonium was photometrically estimated according to the DIN 38406-E5 protocol on the basis of an indophenol blue reaction and measured at a wave length of 691 nm. The phosphate analysis was also performed photometrically according to the DIN 38405-D11-1 protocol based on the reaction of o-phosphate and molybdenum acid to heteropoly acid, phosphomolybdic acid in a sulphuric solution. The latter was detected at a wavelength of 334 nm. The analysis was conducted using an EPOS Analyser 5060 (Eppendorf, Germany) (Fig. 4).

Analogically to the DIN ISO 22036 the concentrations [mg/L] of As, Ca, Fe, K, Na, Mg, Mn, and S were measured with atom emission spectroscopy with inductive coupled plasma (ICP-AES; Spectro analytical instruments, Spectro Ciros CCD, Germany) (Fig. 4).

After 8 weeks *L. perenne* was harvested and microbial biomass (CFE method) was estimated using the chloroform fumigation extraction (CFE) method (Anderson and Domsch, 1978). Reasonable substrate mixtures and fertilization requirements for the Chinh Bac field experiment were determined as result of the pre-experiments (Table 1).

2.2. Chinh Bac field experiment

The Chinh Bac field experiment (Fig. 2) was established as a completely randomized block design in March/April 2009 to investigate different substrate mixtures and plant species for a fast and low-cost restoration of dumps in the open-cast mining area of northern Vietnam. The climate condition is subtropical with two seasons: hot and moist summer from April to October (average temperature 28 °C), and dry and cold winter from November to March (average temperature 16°); with an annual average temperature of 23 °C and a mean annual precipitation of 2000–2500 mm (Nguyen et al., 2004).

The experiment was divided into 6 blocks (6 replications) of 12 plots each, including all possible combinations of the following two

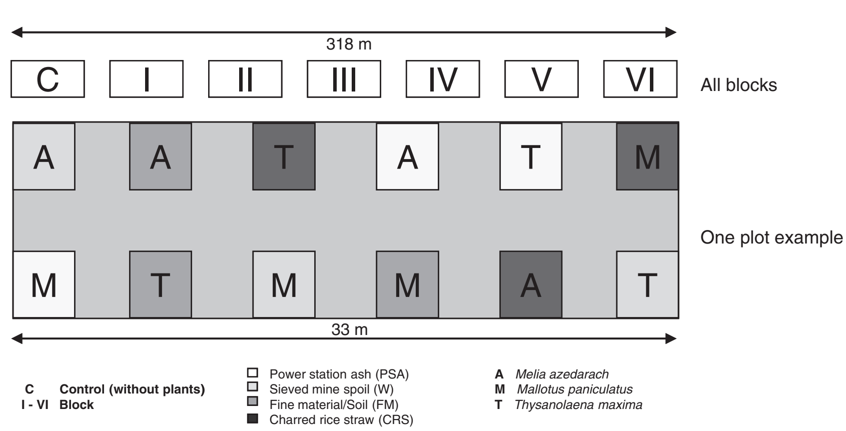

Fig. 2. Scheme of Chinh Bac field experiment.

Table 2
Results of parameter estimation, standard deviation (s) and statistical classes of Chinh Bac field experiment (n = 6).

		Thysanolaena maxima								*Mallotus paniculatus*								*Melia azedarach*							
Method	Date	PSA	s	CRS	s	FSM	s	W	s	PSA	s	CRS	s	FSM	s	W	s	PSA	s	CRS	s	FSM	s	W	s
pH $CaCl_2$	15.03.09	3.52 a	0.42	3.93 a	0.51	3.62 a	0.52	3.68 a	0.47	3.73 a	0.43	3.79 a	0.61	4.48 a	0.50	3.97 a	0.68	3.95 a	0.85	4.23 a	0.60	4.01 a	0.96	4.43 a	1.17
	16.06.09	3.50 b	0.46	5.16 a	1.05	3.39 b	0.16	3.84 b	0.69	3.98 a	0.65	4.40 a	0.94	3.49 a	0.21	4.03 a	0.66	3.98 ab	0.73	5.02 a	1.08	3.44 b	0.23	4.63 ab	1.23
	22.10.09	3.51 b	0.36	4.98 a	1.10	3.38 b	0.22	3.65 b	0.56	3.70 a	0.50	4.11 a	0.82	3.52 a	0.15	4.04 a	0.72	3.88 a	0.67	4.71 a	0.95	3.55 a	0.49	4.49 a	1.17
	09.03.10	3.57 b	0.38	4.68 a	1.00	3.39 b	0.23	3.64 b	0.53	3.58 a	0.32	4.27 a	0.80	3.48 a	0.15	4.03 a	0.71	3.87 a	0.77	4.58 a	0.92	3.46 a	0.30	4.33 a	1.05
	21.09.10	3.53 b	0.45	5.03 a	0.93	3.37 b	0.26	3.75 b	0.57	3.60 a	0.44	3.99 a	0.79	3.40 a	0.20	4.06 a	0.76	3.78 a	0.73	4.43 a	0.82	3.34 a	0.23	4.54 a	1.43
EC [$\mu S\ cm^{-1}$]	15.03.09	339.8 a	137.4	334.5 a	223.3	322.1 a	108.2	323.4 a	124.9	270.1 a	104.7	359.6 a	176.8	422.8 a	271.1	253.2 a	153.2	335.3 a	271.6	315.0 a	163.0	285.9 a	112.7	381.3 a	319.6
	16.06.09	322.5 a	145.5	304.6 a	156.0	309.6 a	114.3	289.6 a	104.8	226.8 a	99.2	330.1 a	164.4	371.0 a	250.3	295.0 a	161.4	302.4 a	267.8	380.4 a	147.9	345.2 a	109.8	340.8 a	258.0
	22.10.09	41.1 a	15.4	101.9 a	78.6	66.8 a	16.8	57.4 a	10.7	63.3 a	29.1	64.4 a	33.0	96.6 a	65.0	60.6 a	27.8	109.0 a	111.8	81.7 a	40.4	68.1 a	27.8	109.4 a	95.9
	09.03.10	66.6 a	11.9	116.7 a	52.2	89.6 a	17.5	80.1 a	26.5	98.2 a	47.1	121.7 a	99.3	205.3 a	187.7	93.4 a	27.3	163.0 a	183.2	111.5 a	53.7	172.1 a	73.4	132.2 a	97.1
	21.09.10	30.9 a	4.2	33.4 a	10.8	37.1 a	7.8	32.3 a	17.4	30.1 a	8.8	37.4 a	17.2	42.3 a	23.8	35.7 a	17.1	49.6 a	41.5	33.8 a	9.7	31.3 a	18.8	36.7 a	26.2
TOC [%]	15.03.09	4.24 a	2.18	4.69 a	1.87	3.52 a	1.63	5.38 a	2.18	5.12 a	2.79	4.16 a	1.93	4.46 a	1.56	5.34 a	2.57	5.14 a	1.66	4.55 a	2.79	3.98 a	0.93	3.21 a	1.65
	16.06.09	4.95 b	2.52	4.33 b	1.60	9.56 a	1.80	4.63 b	1.96	7.20 ab	3.86	4.89 b	2.01	9.55 a	1.32	5.22 b	1.82	5.29 ab	1.53	5.03 b	2.22	10.02 a	3.45	4.00 b	1.93
	22.10.09	5.60 a	3.63	4.39 a	2.14	7.28 a	1.75	4.78 a	1.69	5.74 ab	2.28	3.94 b	0.65	7.47 a	1.34	5.50 ab	2.15	5.69 ab	1.54	5.23 ab	2.57	7.76 a	1.62	3.50 b	1.81
	09.03.10	4.28 ab	1.76	3.53 b	0.95	6.79 a	2.35	5.08 ab	1.82	5.60 ab	2.53	3.29 b	0.89	7.43 a	2.40	5.21 ab	1.94	6.04 ab	1.33	5.06 ab	2.66	7.05 a	0.88	3.53 b	1.21
	21.09.10	5.12 b	1.91	4.66 b	1.53	8.21 a	1.82	5.01 b	2.32	6.14 ab	2.42	3.29 b	1.92	7.97 a	2.50	5.49 ab	2.12	6.63 b	1.94	5.14 b	2.51	7.70 a	3.02	4.07 b	1.46
TON [%]	15.03.09	0.075 a	0.023	0.084 a	0.023	0.083 a	0.044	0.096 a	0.033	0.083 a	0.038	0.069 a	0.023	0.088 a	0.026	0.086 a	0.035	0.088 a	0.018	0.085 a	0.030	0.072 a	0.019	0.070 a	0.039
	16.06.09	0.062 b	0.020	0.083 b	0.018	0.148 a	0.039	0.073 b	0.019	0.102 ab	0.035	0.079 b	0.017	0.131 a	0.016	0.079 b	0.019	0.081 b	0.015	0.081 b	0.016	0.131 a	0.027	0.070 b	0.020
	22.10.09	0.085 a	0.029	0.084 a	0.015	0.107 a	0.036	0.090 a	0.032	0.086 ab	0.014	0.074 b	0.013	0.115 a	0.029	0.094 ab	0.032	0.092 ab	0.018	0.084 ab	0.017	0.100 a	0.007	0.074 b	0.020
	09.03.10	0.091 a	0.026	0.101 a	0.023	0.129 a	0.044	0.103 a	0.045	0.107 ab	0.044	0.080 b	0.033	0.141 a	0.051	0.116 ab	0.043	0.124 ab	0.026	0.103 ab	0.043	0.139 a	0.015	0.091 b	0.027
	21.09.10	0.108 b	0.031	0.120 ab	0.020	0.156 a	0.024	0.117 ab	0.040	0.123 a	0.025	0.094 a	0.032	0.148 a	0.054	0.123 a	0.039	0.134 a	0.029	0.116 a	0.038	0.152 a	0.041	0.105 a	0.029
CECeff [%]	15.03.09	32.0 a	7.5	33.1 a	14.3	29.7 a	3.8	36.0 a	7.5	33.0 a	9.6	37.0 a	18.4	45.8 a	15.8	34.8 a	8.9	36.7 a	14.8	35.3 a	11.2	32.5 a	8.3	51.2 a	38.0
	16.06.09	31.8 b	6.2	46.5 a	10.1	35.4 ab	7.8	33.1 ab	2.7	33.9 a	5.7	40.5 a	12.1	42.4 a	17.1	37.0 a	2.8	40.0 a	13.2	47.5 a	8.5	39.7 a	7.7	45.2 a	18.2

	22.10.09	17.5 b	4.6	28.5 a	6.4	21.3 ab	3.3	21.9 ab	3.8	23.5 a	5.0	28.9 a	16.7	26.7 a	2.7	24.8 a	4.8	26.7 a	4.6	27.1 a	7.1	24.1 a	3.3	25.7 a	6.4
	09.03.10	18.3 b	5.9	25.4 a	5.2	21.4 ab	3.7	22.2 ab	5.7	22.7 a	4.2	23.9 a	8.4	30.3 a	10.3	24.8 a	5.6	28.2 a	6.8	26.4 a	8.9	26.3 a	5.4	24.5 a	5.8
	21.09.10	17.8 b	6.2	26.2 a	7.0	21.2 ab	2.1	23.0 ab	4.7	20.5 a	3.7	21.8 a	5.5	23.3 a	1.8	23.4 a	6.7	23.2 a	4.5	24.4 a	8.1	21.8 a	3.6	22.9 a	6.2
P [mg kg^{-1}]	15.03.09	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.
	16.06.09	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.
	22.10.09	6.223 a	9.267	5.276 a	4.711	5.007 a	4.492	2.029 a	3.196	4.833 a	5.913	1.952 a	3.063	2.407 a	3.851	4.303 a	4.759	2.204 a	3.494	6.221 a	6.203	1.720 a	4.213	4.543 a	3.639
	09.03.10	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.
	21.09.10	n.m.	n.m.	0.842	2.062	0.925	2.266	0.992	2.429	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.
CaO [%]	15.03.09	0.066 a	0.020	0.077 a	0.023	0.065 a	0.007	0.069 a	0.006	0.071 a	0.018	0.076 a	0.027	0.105 a	0.030	0.074 a	0.021	0.082 a	0.035	0.083 a	0.016	0.072 a	0.015	0.092 a	0.041
	16.06.09	0.049 b	0.009	0.088 a	0.022	0.056 b	0.010	0.064 ab	0.015	0.071 a	0.021	0.080 a	0.030	0.069 a	0.018	0.064 a	0.012	0.077 a	0.021	0.079 a	0.020	0.064 a	0.014	0.088 a	0.031
	22.10.09	0.042 b	0.009	0.071 a	0.018	0.042 b	0.008	0.048 b	0.008	0.053 a	0.013	0.057 a	0.019	0.056 a	0.011	0.054 a	0.012	0.060 a	0.015	0.065 a	0.021	0.048 a	0.011	0.068 a	0.024
	09.03.10	0.044 b	0.011	0.068 a	0.018	0.045 b	0.009	0.051 ab	0.010	0.054 a	0.009	0.062 a	0.023	0.065 a	0.021	0.058 a	0.011	0.061 a	0.016	0.064 a	0.016	0.053 a	0.013	0.066 a	0.018
	21.09.10	0.042 b	0.010	0.074 a	0.018	0.043 b	0.007	0.050 b	0.011	0.050 a	0.007	0.052 a	0.016	0.048 a	0.006	0.053 a	0.014	0.054 a	0.013	0.061 a	0.016	0.043 a	0.008	0.062 a	0.023
Fe_2O_3 [%]	15.03.09	1.112 a	0.396	1.598 a	0.378	1.281 a	0.385	1.440 a	0.283	1.542 a	0.284	1.532 a	0.636	2.044 a	0.353	1.704 a	0.611	1.591 a	0.552	1.809 a	0.452	1.495 a	0.342	1.879 a	0.606
	16.06.09	1.243 b	0.301	1.879 a	0.512	1.461 ab	0.312	1.632 ab	0.297	1.574 a	0.437	1.504 a	0.623	1.723 a	0.201	1.628 a	0.486	1.668 a	0.517	1.680 a	0.376	1.513 a	0.226	2.009 a	0.669
	22.10.09	1.219 b	0.359	1.785 a	0.456	1.373 ab	0.260	1.675 ab	0.332	1.474 a	0.407	1.473 a	0.595	1.781 a	0.388	1.641 a	0.424	1.625 a	0.434	1.702 a	0.480	1.489 a	0.337	1.959 a	0.593
	09.03.10	1.368 b	0.352	1.922 a	0.372	1.655 ab	0.322	1.717 ab	0.346	1.747 a	0.260	1.647 a	0.535	2.052 a	0.232	1.827 a	0.447	1.850 a	0.399	1.820 a	0.333	1.775 a	0.264	2.035 a	0.537
	21.09.10	1.397 b	0.380	1.885 a	0.442	1.687 ab	0.187	1.813 ab	0.276	1.695 a	0.310	1.607 a	0.488	2.010 a	0.220	1.800 a	0.485	1.737 a	0.357	1.872 a	0.343	1.755 a	0.267	2.063 a	0.563
Mn [ppm]	15.03.09	56.7 a	18.3	80.8 a	17.0	62.3 a	25.3	71.0 a	12.3	67.2 a	13.8	76.2 a	36.3	114.2 a	18.1	84.5 a	34.7	79.0 a	38.2	98.5 a	33.6	75.0 a	30.8	107.7 a	51.4
	16.06.09	68.7 b	11.7	128.3 a	39.0	69.0 b	16.8	96.2 ab	29.1	107.2 a	48.4	129.3 a	84.9	92.2 a	25.6	87.5 a	26.4	105.7 a	39.1	127.7 a	30.9	82.7 a	17.8	130.5 a	54.8
	22.10.09	48.8 b	18.3	118.2 a	44.3	50.5 b	20.9	66.3 b	23.5	75.7 a	21.6	84.3 a	46.5	69.7 a	15.8	84.0 a	36.4	80.0 a	25.5	116.5 a	49.6	62.5 a	19.8	110.3 a	63.2
	09.03.10	45.5 b	20.0	102.5 a	41.2	51.0 b	21.8	61.5 b	22.5	63.2 a	17.6	87.5 a	51.4	74.7 a	19.2	79.5 a	28.0	77.5 a	31.7	99.0 a	38.0	57.0 a	14.0	91.2 a	40.5
	21.09.10	49.0 b	25.1	136.0 a	37.4	48.3 b	21.4	76.0 b	28.4	65.0 a	25.7	77.7 a	44.8	67.8 a	12.3	80.0 a	36.4	79.0 a	34.1	123.0 a	47.0	55.7 a	18.7	116.0 a	76.9

n.m. — not measurable.
a, ab, b, c — significant statistical classes.

factors — four amendments: a) without (W) as control treatment b) 0.3 M% CRS — according to the results of the pre-experiment c) 0.06 M% PSA — according to the results of the pre-experiment as well and d) 20 M% FSM as common treatment — and three plant species aa) *Thysanolaena maxima* bb) *Mallotus paniculatus* and cc) *Melia azedarach*. The plants were fertilized with an initial application of N–P–K according to 50:100:30 kg ha^{-1} in May 2009. The treatments with CRS and with PSA were selected due to their good results during the pre-experiments. The treatment with fine material represents the current standard as a planting substrate by the Nui Beo coal company with the power station ash originating from the coal power station in Uông Bí. The charred rice straw was produced by carefully and slowly burning dry rice straw and quickly applying water to obviate a complete combustion.

Each plot has a size of 3 m × 3 m and a 0.2 m-thick layer of substrate mixture. The distance between plots amounts to 3 m and between blocks to 20 m. The total length of the experiment area equals 330 m and the width spans 9 m. The in situ mine spoil was sieved through a large sieve with an aperture size of 0.1 m × 0.1 m. After sieving the amendments were manually mixed into the plots.

Substrate samples from all 72 plots were collected on 15 March 2009 before and after the substrate application on 16 June 2009, 22 October 2009, 9 March and 21 September 2010 to take the rainy and dry season into account.

All substrate samples were pre-treated according to the DIN ISO 11464 protocol and sieved to a particle size of 2 mm. The samples were analyzed in terms of pH, electrical conductivity (EC), content of carbonates, total carbon (TC) and total nitrogen (TN), effective cation exchange capacity (CEC_{eff}), base saturation (BS), available potassium (K) and phosphorus (P), and total Ca, Fe, Mn, and S concentrations (Table 2).

Soil pH was determined potentiometrically (METTLER TOLEDO, Seven Easy, Germany) in a suspension with H_2O or 0.01 m $CaCl_2$ at an SSR of 1:2.5 (DIN 19684) (Table 2). To estimate EC according to the DIN ISO 11265 protocol the air-dried soil was extracted with bi-distilled water in an extraction ratio of 1:5. The measurement was performed in the filtered extract with a conductivity analyzer (WTW, Cond 330i, Germany) (Table 2). After determining the concentration of TOC and TON in the soil samples using an elemental analyzer (Vario EL, Elementar GmbH, Hanau, Germany) the soil samples were burned in an oxygen gas stream and the thermal conductivity was measured (Table 2).

In order to determine the proportion of a soil's CEC_{eff} occupied by basic and acidic cations a sub-sample was treated with HCl according to the DIN 19682-13 protocol (Table 2). No carbonates were detectable. The sum of basic cations divided by the CEC_{eff} is the BS percentage. According to Meiwes et al. (1984) CEC_{eff} and BS are estimated with ammonium chloride (HFA 3.2.1.1 Waldböden (BZE) (Fig. 9). The exchange was performed by shaking the samples with 25 ml ammonium chloride solution four times. The amounts of Al, Ca, Fe, K, Na, Mg, and Mn were estimated according to DIN ISO 22036 by CP-AES (Spectro analytical instruments, Spectro Ciros CCD, Germany).

For estimating P and K a double lactate extract was compounded and P and K were measured in a filtered extract by ICP-AES (Spectro analytical instruments, Spectro Ciros CCD, Germany) (Hoffmann, 1991) (Table 2 & Fig. 6).

Total Ca, Fe, Mn, and S concentrations were determined by XRF (WDXRF: S4 Pioneer, Germany and EDXRF: XLAB 2000; Germany) in accordance with DIN EN 15309 (Table 2).

2.3. Statistics

The statistical analysis for pre-experiments was undertaken using the package PASW v.18.0 (STATISTICA).

For all parameter results of the field experiment (6 replications for each parameter) the exclusion of Gaussian distribution were tested with the Kolmogorov–Smirnov test. In every case the Gaussian distribution could not be excluded. Secondly, the data set was tested with Shapiro–Wilks test, to proof the Gaussian distribution too. This test showed a non-Gaussian distribution for half of the data set. In case of a non-Gaussian distribution additionally the logarithms of data were used. The correlations were assessed using Pearson's coefficient. After the one-way analysis of variance, differences among groups were tested using the Duncan test (resulting statistical classes: a, b, c). An error probability level of 0.05 was used throughout the tests as threshold of significance.

The R system version 2.7.1 (R Development Core Team, 2011) together with the 'agricolae' package by de Mendiburu (2010) was applied for further statistical computing. Based on the results of a two- or one-way ANOVA the significance of differences was determined using the test procedures by Tukey, Scheffé Student-Newman–Keuls (SNK) and the LSD test with the usual probability level of 0.05 as the threshold of significance.

3. Results

3.1. Pre-experiments

3.1.1. pH test series

Before starting the pre-experiment the original substrate from the dump site was analyzed by XRF revealing a typical composition for open-cast mining spoil. It was characterized by high mean values of S (1420 ppm), Pb (34 ppm), Ni (60 ppm) and As (50 ppm) and a low value of CaO (0.094%).

In the pre-experiment the effect of different amounts of amendments a) PSA and b) CRS on the pH value of dump substrate was investigated as a test series. A linear relationship exists between the input amount of both amendments and the dump substrate that can be summarized by the following two equations, the first representing PSA and the second CRS.

$$pH_{PSA} = 20.42\,PSA + 5.20(n = 4, R = 0.96) \quad (1)$$

$$pH_{CRS} = 2.17\,CRS + 5.19(n = 7, R = 0.86) \quad (2)$$

with PSA and CRS in M%.

Eqs. (1) and (2) represent that the increase of the pH value with added PSA is much higher than with CRS. The aim of this pre-experiment was to define the amount of amendments required to almost neutralize the dump substrate.

A pH value of 6.52 was measured with an input of 0.06 M% PSA. This value served as the base amount for the climatic chamber

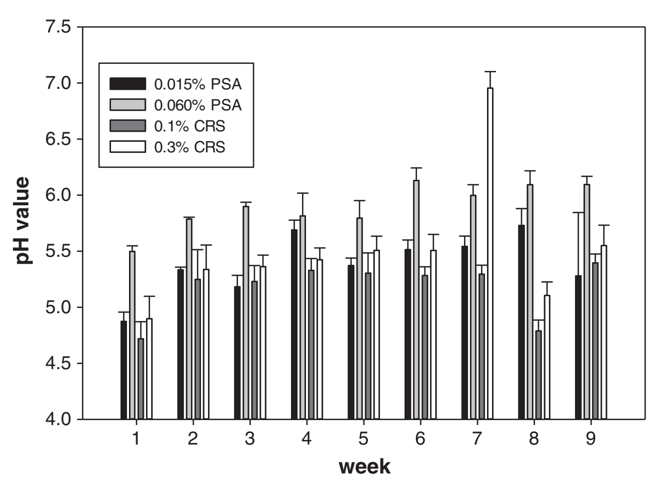

Fig. 3. Development of pH value with standard deviation in climatic chamber experiment.

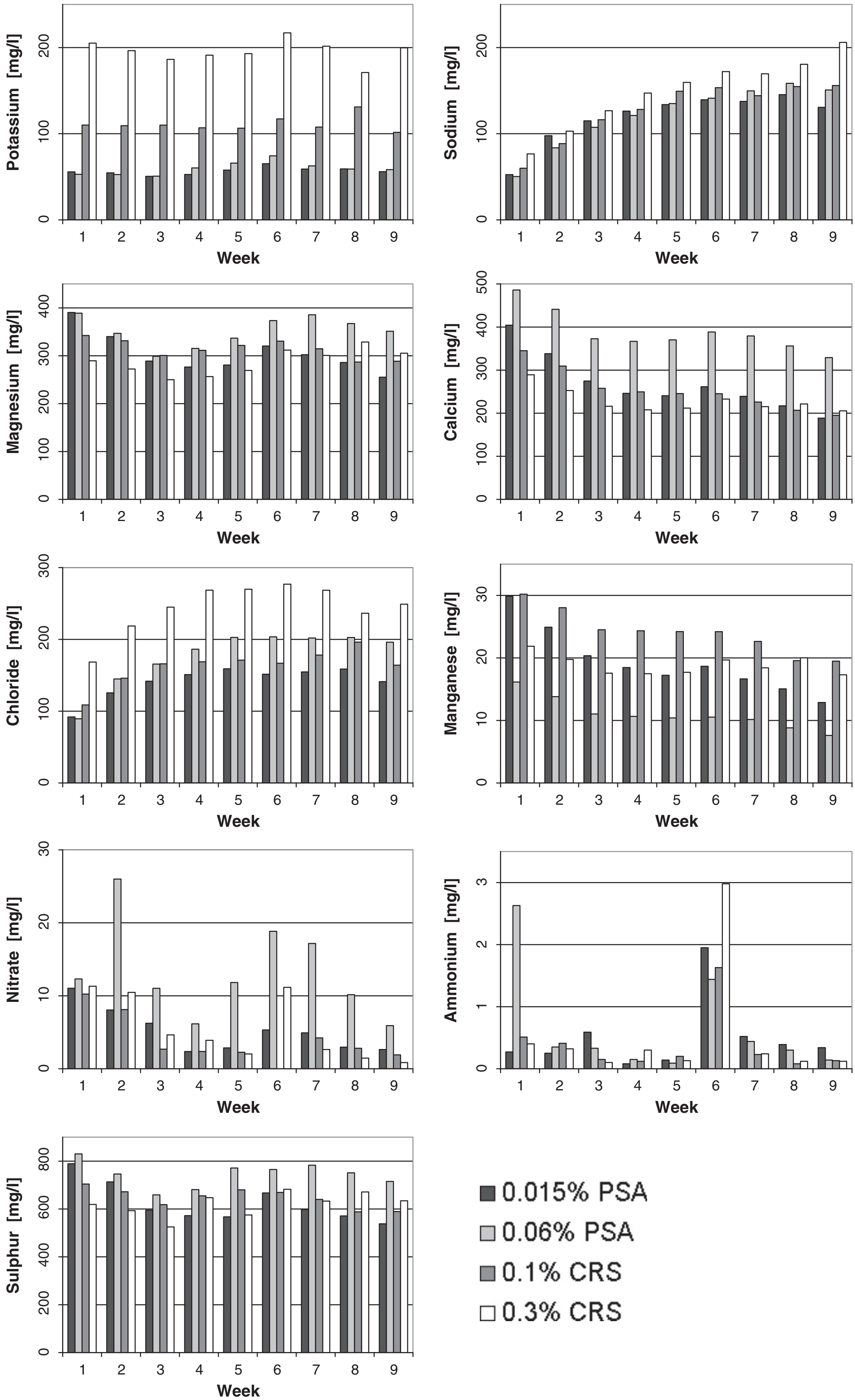

Fig. 4. Development of soil solution parameters in climatic chamber experiment.

experiment. The addition of 0.015 M% PSA was chosen to test a lower pH increase effect on plant growth as the second treatment for this experiment. It was determined that the required input of amendment to achieve pH neutrality for the treatment with CRS is not compatible with a practicable application rate in situ. Based on these facts two treatments with 0.3 M% and 0.1 M% CRS were investigated in the climatic chamber experiment.

3.1.2. Climatic chamber experiment

The pH values ($n = 3$) of each soil substrate mixture show an increase from the initial value of pH 4.9 over the experiment duration of nine weeks (Fig. 3). At the beginning of the experiment the ph increase in PSA treatments is higher than in the CRS treatments. A lower pH value increase was measured respectively for both treatments with a lower input of amendments, 0.015 M% PSA and 0.1 M% CRS. However, the pH value increased more over time in the treatment with CRS than in the treatment with PSA, although the pH values were always higher in the treatments with PSA, except the pH value of the treatment with 0.3 M% CRS in week 7.

The investigation results of the soil solutions with different substrate mixtures are represented in Fig. 4. It is known that the ions of potassium, sodium, magnesium, and calcium affect the base saturation (BS) and the pH value when allocated to the exchange places. The amount of potassium was found to be much higher in the CRS treatments than in the PSA treatments due to the rice straw input. Also the amount of sodium ions, accompanied by chloride ions (corr. Na/Cl 0.74 for $p < 0.05$ and $n = 4$), is higher in the CRS treatments than in the corresponding PSA treatments. On the other hand the amount of calcium ions, accompanied by magnesium ions (corr. Ca/Mg 0.79 for $p < 0.05$ and $n = 4$), is significantly higher in the PSA treatments than in the CRS treatment.

The amounts of nitrate and ammonium in the soil solution are very low. Increased nitrate amounts were only measured in the 0.06 M% PSA treatment.

The highest values of S were measured in the 0.06 M% PSA treatment at all times. The further ranking was unclear. At different sampling dates the second highest level of S in the soil solution was observed in different treatments.

The measured values of plant-toxic arsenic in soil solutions were always under the detection limit as were mostly the values of phosphorus, which is very important for plant nutrition.

The growth of *L. perenne* in the experiment was very poor. However the results of harvested plant compounds (see Table 1) represent higher amounts of below- and above-ground biomass in the CRS treatments in comparison with the application rates of PSA. The measured microbial biomass was higher in the treatments with a higher application rate, although the difference between the amendments was negligible. The field experiment was conducted to gain a better insight.

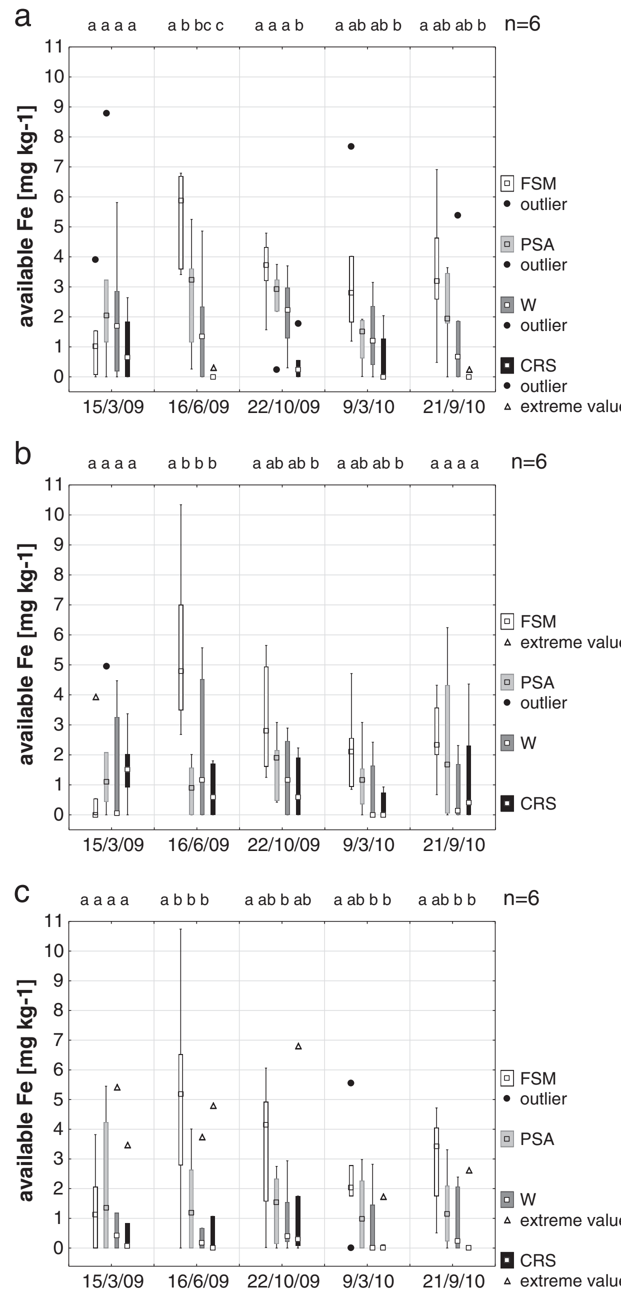

Fig. 5. Box–Whisker Plot with and statistical classes of available Fe in Chinh Bac experiment. (a) *T. maxima*; b) *M. paniculatus*; c) *M. azedarach*.

3.2. Chinh Bac field experiment

As shown in Table 2 and in Figs. 5 to 10 the chemical conditions of all treatments of investigated spoil before applying the amendments on 15 March 2009 were similar. None of the investigated parameters revealed significant differences between the treatments.

After admixing the amendments on the substrate sampling date, 16 June 2009, significant changes of chemical conditions in all treatments were observed with regards to TOC (Table 2), TON (Table 2) and available Fe (Fig. 5) for FSM treatments and to available K (Fig. 6) for CRS treatments, respectively. The reasons for these significant changes of TOC and available Fe for the FSM treatments in comparison to all other treatments was the input of anthracite residuals accompanied by pyrite (FeS_2), contained in the FSM (see Table 2 and Fig. 5). After applying the amendments marginal increases of TOC contents were also visible in PSA and CRS treatments whereas a larger C/N ratio did not affect an increase of TON. The increase of TOC content in the PSA treatment was caused by coal particles contained in the power station ash. A higher increase of TOC content than measured was expected in the CRS treatment which was enriched with organic C from charcoaled rice straw. The effect, however, was marginal. The initial value of C in spoil is much higher than the C input originating from rice straw. Due to an unchanged C/N ratio compared with the other treatments the increase of TOC content in CRS treatment also leads to an increase of TON content after substrate mixture on the second sampling date (Table 2). This was also reflected by a correlation coefficient of 0.77 between TOC and TON. During the experiment the mean value of TON content in the FSM treatments was always higher than in the other treatments yet the difference to other treatments was not always significant. After 22 October 2009 TON contents in all treatments were increased between 0.02 and 0.05% (Table 2).

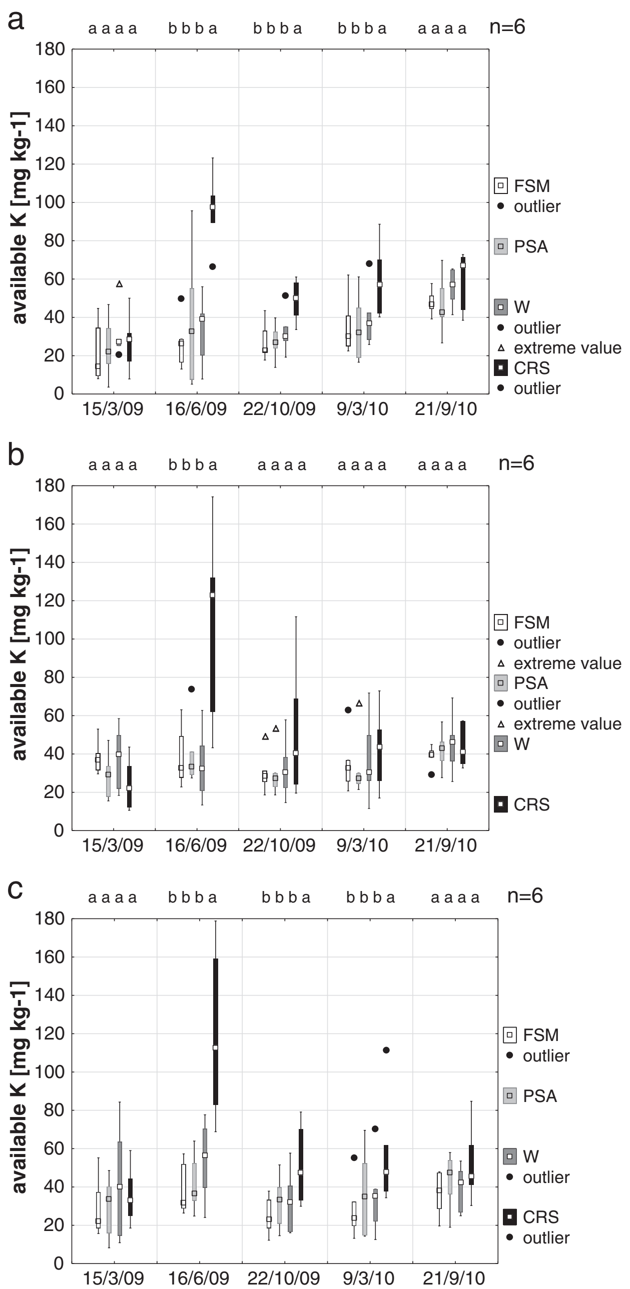

Fig. 6. Box–Whisker Plot with statistical classes of available K in Chinh Bac experiment. (a) *T. maxima*; b) *M. paniculatus*; c) *M. azedarach*.

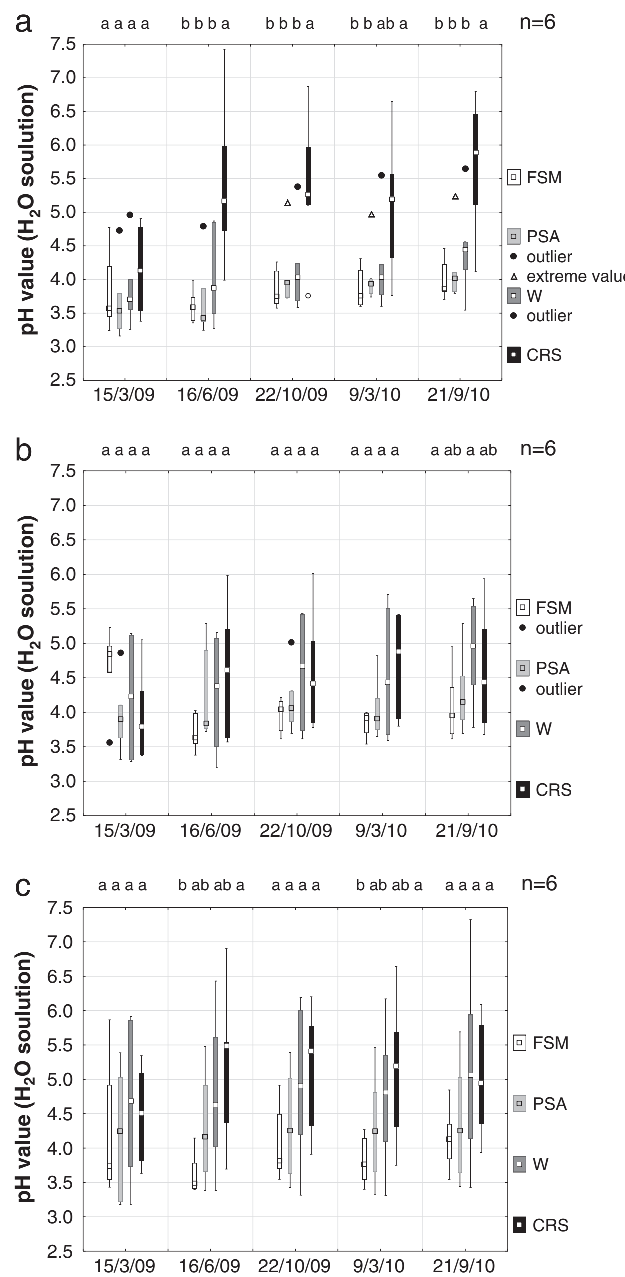

Fig. 7. Box–Whisker Plot with statistical classes of pH value in H_2O solution in Chinh Bac experiment. (a) *T. maxima*; b) *M. paniculatus*; c) *M. azedarach*.

The significant increase of available K for all plant CRS treatments is explainable by high K contents of rice straw (Fig. 6). Significant higher contents of available K in the CRS treatment only exist in plots with *M. paniculatus* on the second sampling date and for plots with *M. azedarach* and *T. maxima* up to the fourth sampling date (Fig. 6). At the end of the experiment no considerable differences between the treatments could be observed.

Most other significant differences between the treatments are strongly plant specific to *T. maxima* (Table 2, Figs. 5–10). *T. maxima*, a fast-growing grass, had a high input of organic material to the soil.

All substrate samples showed an acidic reaction (Fig. 7 and Table 2) regardless of which method was applied. The pH value which is measured in an H_2O extract is generally higher than the pH in a $CaCl_2$ extraction for all treatments. Hence, there are primarily cation exchangers and in addition primarily H^+ ions are bound by the exchangers in all substrate mixtures. This leads to the assumption that humic substances are missing. A noticeable increase of pH (H_2O, Fig. 2 and $CaCl_2$ solution, Table 2) over time was established for the CRS treatment of *T. maxima* plots. Besides available K this increased pH value is accompanied by significantly higher amounts of the available basic cation Ca. (Fig. 8). These cations also affect the BS (Fig. 9) which is substantially higher in the CRS treatment of *T. maxima* plots compared to the other treatments. A similar pattern was also found for CEC_{eff} (Table 2). Significant lower amounts of available Fe and Al were detected (Figs. 5 & 10) as a result of higher pH values in the CRS-treated *T. maxima* plots. The highest values of available Al besides available Fe were measured in the FSM treatment in combination with *T. maxima* and *M. azedarach*. The mean values of available Al in the FSM treatment for *M. paniculatus* plots are slightly increased but not much different to other treatments.

Further significant differences to other treatments of *T. maxima* plots exist in terms of measured Mn, CaO and Fe_2O_3 by XRF

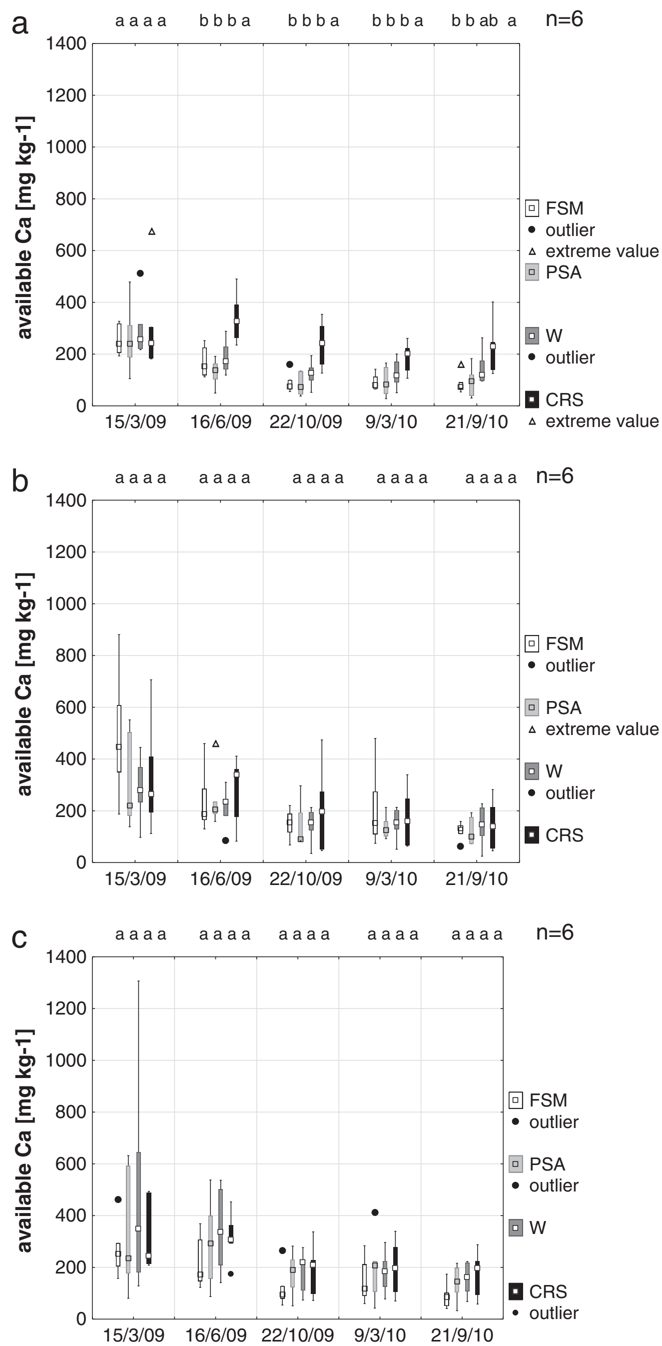

Fig. 8. Box–Whisker Plot with statistical classes of available Ca in Chinh Bac experiment. (a) *T. maxima*; b) *M. paniculatus*; c) *M. azedarach*.

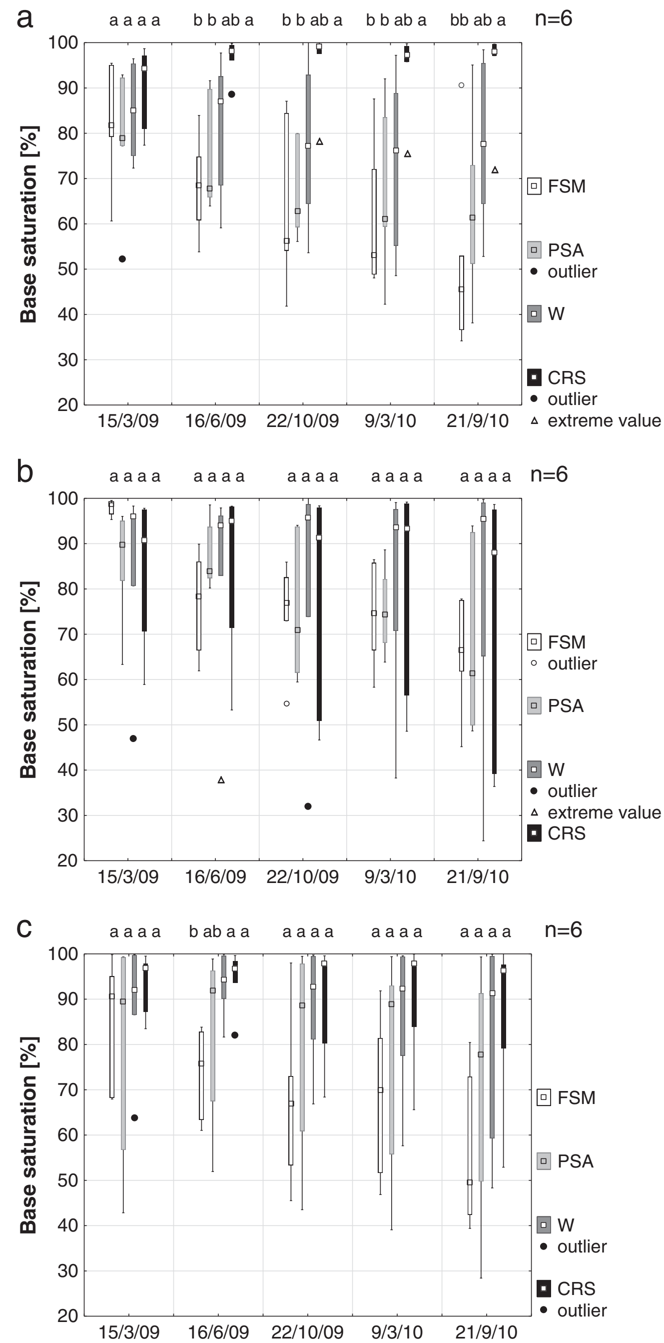

Fig. 9. Box–Whisker Plot with statistical classes of Base saturation in Chinh Bac experiment. (a) *T. maxima*; b) *M. paniculatus*; c) *M. azedarach*.

(Table 2). The values, determined in the CRS treatment after substrate mixture, were always higher than the values in other treatments of *T. maxima* plots. Despite higher values of Fe_2O_3 in this treatment, considerably lower values of available Fe were forecast. Also the values of Mn (XRF) do not correlate with available Mn.

The determined S decreased over time in all treatments. After applying the amendments more S was measured in the FSM treatment whereas only the increased value of FSM in combination with *M. paniculatus* was significant.

In general the measured EC concentrations were very high and did not noticeably differ between the treatments (Table 2). They were similar or even slightly increased on the second sampling date. Up to the third sampling date and after one rainy season the EC measured in all treatments had dropped by more than a third of the previously measured values. Between the third and fifth sampling date a seasonal trend was visible. The measured values of EC after the rainy season (22 October 2009 & 21 September 2010) were lower than the values measured after the dry period (9 March 2010).

In all plots the contents of plant available P were very low (Table 2). Despite the NPK fertilizer added between the first and second sampling date no available P was measurable on the second sampling date. P was only measurable in all treatments on the third sampling date. One possible explanation is the time required for the fertilizer to spread in spoil during the rainy season. In turn no P was detected on the fourth sampling date. Plant-derived P was definitely expected in CRS, W, and FSM in combination with *T. maxima*.

The highest negative correlations ($p < 0.05$, $n = 360$) consist between BS and available Al (0.91). Especially positive correlations

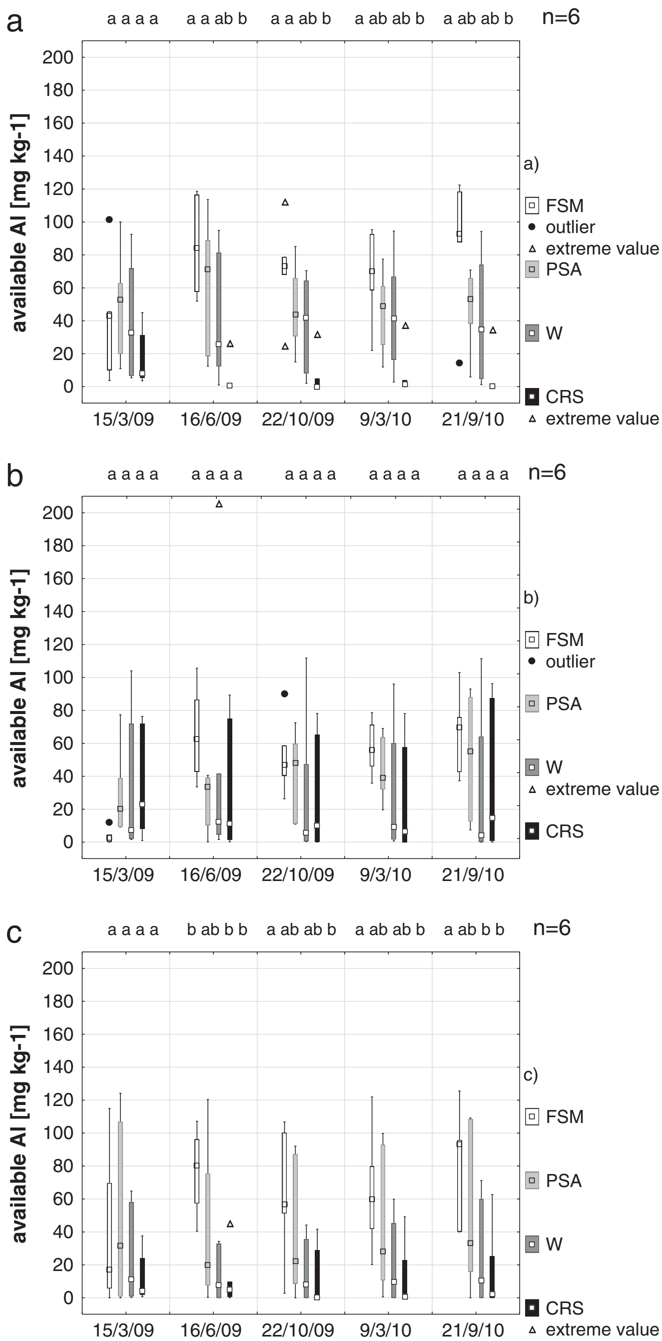

Fig. 10. Box–Whisker Plot with statistical classes of available Al in Chinh Bac experiment. (a) *T. maxima*; b) *M. paniculatus*; c) *M. azedarach*.

($p < 0.05$, $n = 360$) were found between CEC_{eff} and available Ca (0.9)/CaO (0.8) as well as available Mg (0.9). Furthermore positive correlations ($p < 0.05$, $n = 360$) existed between pH $CaCl_2$ and Mn (0.83), BS (0.73) plus available K (0.66). On the other hand low pH $CaCl_2$ values cause high values of available Al (corr. − 0.75) and available Fe (corr. − 0.65). High correlations ($p < 0.05$, $n = 360$) were also found between available Mg and available Ca (0.80); available Fe and available Al (0.77); Mn and available Mg (0.70).

4. Discussion

The results of the pre-experiments allowed the amount of amendments necessary to enable plant growth for different plant types concerning pH sensitivity to be calculated (Fig. 3). However it is extremely necessary to analyze the amendment which is applied in the field in respect to its actual chemical composition. The power station ash (fly ash from coal power station in Uông Bí) used in Vietnam did not have the same effect on the soil chemical processes as the power station ash tested in the pre-experiments. We were forbidden to carry out investigations of the German PSA (requirement of coal power station). In Vietnam it was not possible to test the PSA in situ before setting up the field experiment due to missing laboratory equipment.

Furthermore the results of the climatic chamber experiment indicated problematic chemical conditions for plant growth and proved that the spoil is nutrient-poor (Fig. 4). Seeded or planted vegetation requires sufficient nutrients to restore dump sites. Despite a stronger increase of pH values in PSA treatments in the climatic chamber experiment, the plant growth of shoots and roots in CRS treatments were much better than in comparable PSA treatments (Table 1). A possible reason is the higher availability of K as a plant nutrient. The limitation of 12 places in the climatic chamber has obviated the control variant. But this experiment was carried out as pre-experiment to study the effect of different amounts of different amendments in more qualitatively way. The results of climatic chamber experiment have shown that the higher amounts of amendments are necessary to achieve appreciable effect on spoil properties (Fig. 4). Therefore not a fully statistically evaluable experimental design was utilized.

The main result of these field investigations was the realization that CRS in combination with an input of organic matter by root exudates and litter fall as in the case of *T. maxima* ameliorated the spoil to soil-like conditions. Thereafter, CRS mixed with compost, sludge or other easily-available organic materials should be able to affect the substrate quality faster for tree planting. Another possibility is seeding smaller grass types than *T. maxima* and planting trees after one vegetation period. Only the plots of *T. maxima* enriched with CRS differ regarding chemical composition in comparison to other treatments (Figs. 5–10, Table 2). At the end of the experiment the amount of available Al – which is toxic in high doses – strongly decreased (Fig. 10). The same effect was also observed for the available Fe (Fig. 5). CRS increased the pH value effectively and thus reduced the solubility of these ions (Fig. 7, Table 2). Additionally, CRS acted as an activated carbon which can bind ions. Furthermore Mn and Fe_2O_3 in the CRS treatment of *T. maxima* plots are enriched (Table 2), more plant nutrients (Ca and K) are available (Figs. 6 & 8), and the CEC_{eff} and BS are increased (Table 2, Fig. 9). Masulili et al. (2012) gained similar results. However in our investigation we verified that the input of CRS in combination with organic matter has a lasting effect on chemical conditions despite sub-tropical weather conditions characterized by heavy rainfalls.

The poor application effect of mineral NPK fertilizer has shown that the necessary plant nutrients are not available long enough. Due to the high precipitation during the rainy season and the missing humus the nutrients were quickly washed out. In opposition to the other plant covers, the treatment with *T. maxima* plots produced a humus horizon during the course of the experiment.

The benefit achieved from sieving the spoil material is of greater importance than the time advantage gained from the fast planting in FSM. Besides the improved substrate conditions for the planting process, sieving the spoil material accelerated the turnover processes. At the same time this positively influenced the chemical and physical properties of the spoil. In the FSM variant a low pH of approximately 3.5 and high sulphur content from the second sample date indicated an intensive pyrite weathering. Consequently, there was a substantial influence of the fresh fine material on the chemical composition of the spoil that negatively affects plant growth. The acidic pH value caused a higher availability of Al and Fe. Hence, the only advantage of this material was its sand-like texture that is easy to handle at dumps and facilitated the planting.

5. Conclusion

In summary, we have demonstrated that sieving spoil and CRS amendments can serve as a suitable spoil amendment able to sustainably modify the chemical conditions. The strongest influence on spoil properties was observed in the CRS treatment planted with *T. maxima*. To gain a better understanding of the plant properties and their interaction with CRS amendments concerning the impact on dump substrate chemistry more experimental investigation is needed. The selected PSA from the coal power station in Uông Bí was not effective and the FSM amendment led to negative influences on spoil properties.

However, based on these findings it can be ascertained that applying CRS in combination with fast-growing plant species or organic matter is a sustainable possibility of restoring dumps even under sub-tropical conditions with high precipitation in the rainy season which reduces the chemical impacts of additives on the spoil substrate, such as the long term pH increase of PSA. The main requirements for restoring dumps are a sustainable nutrient availability and a reduction of phytotoxic acting ions.

The plant selection for revegetation in terms of salt stress tolerance must be related to the higher EC value during the dry season. However, an additional paper will focus on the plant problematic.

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