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Release dynamics and plant availability of POLY4 fertilizer nutrients in tropical acidic soils

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POLY4 is a polyhalite-based mineral fertilizer containing essential nutrients for crop growth and development. Dissolution of POLY4 and its nutrient availability to crops in acidic tropical soils are currently deemed inadequate. The objective of this study was thus to evaluate the fertilizers' release dynamics of nutrients under controlled conditions, using an open leaching system in the laboratory and in a shade house with *Napier* grass as the test crop. In the open leaching system, Gondang, Selangor and Tawar soils were packed in soil columns similar to their bulk density in the field. Fertilizers were then added at a rate of 100 kg K_2O/ha. Subsequently, the soil columns were leached with distilled water for approximately 30 days, and the nutrients present in the leachate measured. *Napier* grass was planted in polybags filled with soil and treated at 0 and 100 kg K_2O/ha for 280–627 days. The plants were subsequently harvested, and the dry weights recorded, and nutrient uptake calculated. The quantity of K_2O released from POLY4 was greater from the POLY4 standard or chipped (POLY4S) versions than from the POLY4 granule (POLY4G) version. The values ranged from 135.2 µg K_2O, with a pore volume (PV) of 8.4, for the Tawar soils to 46.3 µg K_2O, with a PV of 11.4, for the Gondang soils. Based on nutrient uptake by the *Napier* grass, for each unit of K_2O release, the efficiency of Mg uptake (80.8%) was greater than that of Ca (32.5%). This result is in agreement with a common Mg release pattern as in an open leaching system, suggesting that Mg is an important factor in fertilizer formulation when POLY4 is used for crop production.

Keywords Polyhalite, POLY4 granule (POLY4G), POLY4 standard (POLY4S), Acidic tropical soil, Open leaching system, Dissolution, Plant availability

Potassium (K) and other macronutrients, such as nitrogen, phosphorus, calcium, magnesium, and sulfur are very important nutrients in agriculture. Globally, in 2023, potash consumption exceeded 70 million metric tons, which encompasses potassium chloride, potassium sulfate and potassium nitrate[1]. Potassium chloride accounts for about 90% of all the K sources available globally. Potassium acts as a fundamental driver in crop yield, quality, and resilience against any adverse conditions. In view of this, K management in acid tropical regions is particularly crucial due to the inherent soil limitations and high nutrient loss from heavy rainfall experienced.

POLY4 is a polyhalite-based mineral fertilizer with the chemical formula $K_2SO_4.MgSO_4.2CaSO_4.2H_2O$; and is perceived as slow-release fertilizer. POLY4 contains multiple elements, namely potassium (K) (14% K_2O), magnesium (Mg) (6% MgO), calcium (Ca) (17% CaO), and sulfur (S) (19% S). These are essential nutrients for crop growth and development. The application of POLY4 to crops, therefore, not only supplies K but also supplies Mg and other important nutrients. Additionally, Ca plays an important role as a soil amendment and so the application of POLY4 will certainly contribute to improvements in soil conditions, which could be beneficial for nutrient uptake. These properties of POLY4 could offer better benefits to crops under tropical acid soil conditions as opposed to traditional K sources, such as muriate of potash (MOP), which are highly soluble and prone to leaching. Furthermore, MOP contains Cl^- ions in its chemical formula in contrast to POLY4.

Oil palm is a major tropical crop grown in many acid tropical regions of the world. Currently, MOP (KCl, 60% K_2O) is the main source of K for the **o**il palm crop in major plantations in Southeast Asia, especially Malaysia and Indonesia. Matured oil palm plantations cover about 89.2% of the overall planted area in Malaysia. Large quantities of fertilizers are required annually to sustain high crop yields in these plantations. Further, oil palm

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trees require more K and Mg for yield production; potassium is particularly important for fruit formation[2–4] and increasing the oil content within the fruit's mesocarp[5,6], while magnesium is essential for photosynthesis, as it is a key component of chlorophyll[7–11], and it also affects oil quality.

POLY4 can potentially supply all these 4 nutrients to the oil palm trees. This could help reduce the overall fertilizer costs incurred and increase efficiency in palm oil production. The properties of the MOP fertilizer have been very well studied, and it is thus used in this study as the 'control' for comparison.

Several factors affect the dissolution and nutrient availability of fertilizers, namely, soil characteristics, fertilizer characteristics, crop characteristics and environmental factors. Since POLY4 is a new product in the region, evaluating its dissolution and availability of nutrients to crops is important. Previous studies on polyhalite (< 0.15 mm particle size) only focused on the crop Sorghum-sudangrass (*Sorghum bicolor*, L. Moench 'NB280S—S. Sudanese (Piper) Stapf.) that was grown in Red Feather loamy (Lithic Cryoboralf) sandy soil[12]. Subsequent study by Yermiyahu et al.[13] investigated the solubility of granular polyhalite (< 2.0 mm particle size), under laboratory and field conditions, using desert sandy soil without crop. These investigations however, did not look into the release dynamics of nutrients from POLY4 in acid tropical soil. The objective of this study was thus to evaluate POLY4 nutrient release in the laboratory and in a shade house, using Napier grass (*Pennisetum purpureum*) as the test crop. This investigation is a preliminary study to evaluate the potential of POLY4 in releasing nutrients and its availability in the short term before it is used on long term plantation crops, such as on oil palm trees. Under laboratory conditions, an open leaching system can provide an estimate of the amount of nutrients released[14,15] from fertilizers in the shortest time possible as compared with that under real field conditions, where climate, crop characteristics, and field management have major impacts on nutrients. To validate this fundamental study, a subsequent experiment, using *Napie*r grass as the test crop, was implemented in a shade house. Shade-house conditions, the presence of a test crop and a well-controlled environment can potentially eliminate the varied factors that influence dissolution and nutrient availability to crops. Similar studies have been previously carried out for the evaluation of phosphate rock (PR) materials in the laboratory and under glasshouse conditions[14,15].

Materials and methods

Soils

Three soil series were selected for this study from areas under the management of the Federal Land Consolidation and Rehabilitation Authority (FELCRA): (i) peat soil, Gondang series: Dysic, isohyperthermic, Typic Haplohemist from FELCRA, Simpang Renggam, Johor, 1° 46′ 20.5″ N and 103° 19′ 47.71″ E, 160 m ASL; (ii) saline soil, Selangor series: a fine, mixed, isohyperthermic, Fluvaquentic Dystrudepts from FELCRA, Sedili Besar Estate, Peringkat 5, Johor, 01° 57′ 16.5″ and N 104° 06′ 00″ E, 9 m ASL; and (iii) sandy soil, Tawar series: a fine, mixed, isohyperthermic, Typic Paleudults from FELCRA estate, Sembrong, Johor, 02° 16′ 59.2″ and N 103° 30′ 20.3″ E, 42 m ASL. All these FELCRA estates are in Johor, which is located approximately 400 km to the south of Kuala Lumpur (Fig. 1).

Fertilizer used

POLY4 was supplied in two forms: POLY4-granule (POLY4G) and chipped POLY4-standard (POLY4S). The POLY4G, POLY4S and muriate of potash (MOP, 60% K_2O), as a control, were the fertilizers used in this study.

Simulation of nutrient release from fertilizers in an open system

Leaching columns comprised polyvinyl chloride (PVC) pipes of 5.5 cm, which were cut to 30 cm in length. The selected soil samples (< 2.0 cm particle size) were packed into the soil columns to natural bulk density, similar to that in the field. A rubber stopper fitted with a tubing outlet was fixed to the bottom of each of the soil columns in preparation for the experimental leaching setup[16]. The soil columns were equilibrated on a rack after allowing water to flow up by capillary rise through an outlet at the bottom of the soil columns to saturation point and then allowed to drain completely. The remaining volume of water available in the soil columns represents the pore volume (PV) of the soil. The top 2 cm of soil in the columns was removed and the fertilizers mixed thoroughly with the soil at a rate of 100 kg K_2O/ha. Three soil columns were set up accordingly, representing the Gondang, Selangor and Tawar soil series, to evaluate the release of K_2O from POLY4S and the control. To understand the differences in the release of nutrients from POLY4 as compared to the standard chemical fertilizer, leaching soil columns were set up using the Tawar soil series to which was added an equivalent of 100 kg K_2O/ha. An equivalent quantity of K_2O as that in POLY4 was used for K^+ and Cl^- ions release from Muriate of Potash (60% K_2O); Ca^{2+} and Cl^- ions from Calcium Chloride dihydrate (37.53% CaO; 48.2% Cl^-); and Mg^{2+} and SO_4^{2-} from Magnesium Sulfate heptahydrate (16.37% MgO; 38.94% SO_4^{2-}). The control leaching column was represented by soil only. Then, both of the leaching soil columns were leached with distilled water (pH 6.5–7.0) using a proportioning pump with tubing (Tygon O/Y) at a flow rate of 0.16 mL per minute, and then water allowed to drop on filter paper placed on the soil surface in the top of column. This procedure has been used successfully to measure the dissolution of phosphate rock (PR) materials (PR)[14,15] and phosphogypsum organic (PG organic) in sandy BRIS soils in an open-leaching column[16]. The tops of the columns were covered with waterproof tape.

Leaching of the soils treated with fertilizers continued for up to 30 days, approximately equivalent to the total rainfall for 1 year (2400 mm) in the region. The leaching solutions from the different soil columns were collected and stored in a plastic container for further analysis. The volumes of leachate collected for each column were recorded, with values ranging from 1365.0 to 3561 mL (PV from 4.56 to 12.63).

Sodium, K, Ca and Mg present in the leachate were analyzed using a PinAAcle 900 T atomic absorption spectrometer (AAS) (Perkin Elmer, Shelton, USA). Further, Caand Mg in solution were also determined using AAS, after the addition of 1000 mg Sn/L to reduce spectral interference in the flame mode. Sulfate and Cl^- ions

Fig. 1. Peninsular map showing the three soil sampling sites: Gondang, Selangor and Tawar[31]. *Note* Scale of map; 2.50 cm = 100 km.

in solution were processed with an 858 Metrohm Professional Sample Processor and measured with an 882 Metrohm Compact IC Plus instrument (Ionenstrasse, Switzerland).

Shade house trial

The trial was conducted in the shade house of the Institute of Tropical Agriculture and Food Security (ITAFoS), Integrated Farm, Farm 10, Faculty of Agriculture, Universiti Putra Malaysia (UPM), Serdang, Selangor (GPS 2° 59′ 25.98″ N, 101° 42′ 49.52″ E) (shown in Fig. 2).

Experimental procedure

In this experiment, a tropical grass, *Napier*, was used as the test crop. *Napie*r grass is a perennial plant, where its top portion can be harvested continuously without disturbing the root system of the plants. Its root systems are fibrous and very efficient at extracting and absorbing available nutrients in the soil. Thus, the quantity of elements in the *Napier* plant can be used to estimate the POLY4 fertilizer's dissolution and supply power to the soil. This grass also has considerable potential for use as animal feed in Malaysia (fodder crop).

Planting: treatments and experimental design

One hundred eighty-nine 10-kg soil samples (18 treatments × 3 soils) of air-dried soils were weighed and placed separately into black perforated polybags (26.0 cm wide × 46.0 cm high). To prevent leaching of the nutrients, all the soil filled polybags (inner) were placed inside transparent polybags (outer; 51.0 cm wide × 30.0 cm high), where the height was maintained at ¾ of the inner polybag (Fig. 3). Three *Napier* grass cuttings with 3 nodes (approximately 15.0 cm) were planted in the polybags. They were left to grow for approximately 2 months before being harvested, and the dry weights measured, for the soil homogeneity test.

Fig. 2. Location of the experimental site at the Integrated Farm, Farm 10, Faculty of Agriculture, Universiti Putra Malaysia[31]. *Note* Scale of map; 7.0 cm = 0.2 km.

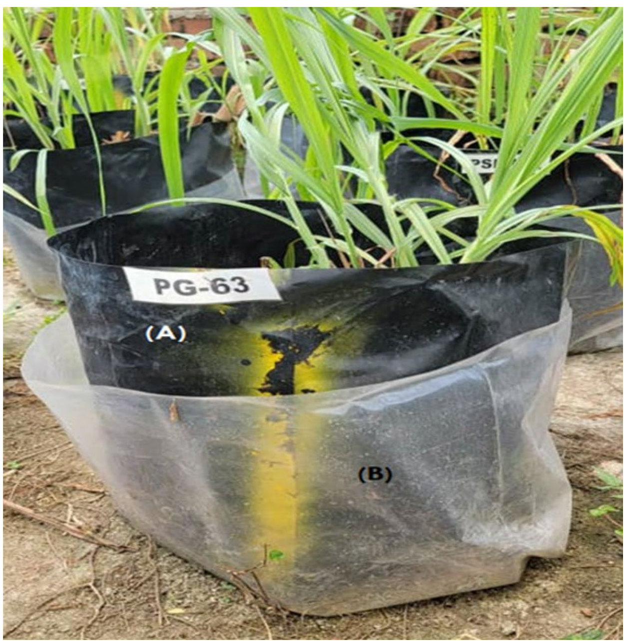

Fig. 3. A typical example of *Napier* grass growing in a polybag (**A**, inner; **B**, outer) in a shade house.

For the soil homogeneity test, the treatments were arranged in a complete randomized design (CRD) with three replications for each soil type. The following levels of K, 0 and 100 kg K_2O/ha, were used for POLY4 fertilizers (14% K_2O), and muriate of potash (MOP; 60% K_2O) was used as the control fertilizer. Distilled water was used to water the soil filled polybags and the soil moisture content was maintained at field capacity. The temperature in the shade house was maintained within the range 28 to 32 °C.

At the appropriate time, when the *Napier* grass was fully established, N at 100 kg/ha (urea, 45% N) and 115 kg P_2O_5/ha (mono ammonium phosphate (MAP), 61% P_2O_5; 12% N) were added in solution form. The addition of MAP was to eliminate common ions, such as that present in POLY4.

Harvesting: plant and soil sampling

The grass was harvested at 5.0 mm above the soil surface to facilitate regrowth, oven-dried, ground to pass through a 2.0 mm sieve and stored in labeled self-adhesive plastic bags for further analysis. Approximately two days after harvest, N and P_2O_5 fertilizer solutions (10.0 mL) were added to all the polybags, to provide an unlimited supply of N and P for good growth of the grass.

At the end of the experiment (harvest 5: 4th after DAT), soil samples were taken at four equidistant sampling points within each polybag using a stainless-steel hand auger (id = 15.0 mm internal diameter), to a depth of 120 mm. The soil samples were then bulked, air-dried, ground, and sieved to pass through a 2 mm sieve. The soil samples were then stored in labeled self-adhesive plastic bags for further analysis.

The work schedule adhered to throughout this study is given in Table 1.

Methods of soil and plant analysis

Methods of soil analysis

The soils used in this study were characterized using standard methods of chemical analysis. Briefly, total C, N and S in the soil were measured using a TruSpec CNS analyzer (LECO, Saint Joseph, MI, USA). The soil pH was measured at a ratio of 1:2.5 using a pH meter (Metrohm 827, Riverview, FL, USA)[17], and the electrical conductivity was measured in saturated gypsum extracts[18] using a conductivity meter (Mettler Toledo SevenEasy™ Conductivity Meter S30, Hamilton, New Zealand).

Organic carbon was measured using the Walkley and Black method[19]. Cation exchange capacity (CEC) was determined by leaching with 1 M NH_4OAC (pH 7.0) solution and displacement of NH_4^+ ions with 1 M K_2SO_4 solution[20]. Soil extractable macro-nutrients were measured using Mehlich 3 extraction solution[21]. The chloride in the soil was extracted by mixing the soil with deionized water at a 1:2 ratio (soil/solution)[22], and the chloride in the solution was determined using the ferricynide colorimetric method[23].

Determination of extractable elements in soil

The presence of metal ions in the soil, as indicated by X-ray fluorescence (XRF), was determined using the Mehlich No. 3 method[20]. The soil sample was extracted at a ratio of 1:10 (soil/solution) using an extraction reagent (containing 0.2 N CH_3COOH, 0.25 N NH_4NO_3, 0.015 N NH_4F, 0.13 N HNO_3 and 0.001 M EDTA) in plastic vials which were shaken for 5 min on a reciprocating shaker at 200 rpm. The solutions were filtered immediately, and the filtrates collected for elemental determination. The elemental concentrations in the solution were measured using inductively coupled plasma-mass spectrometry (ICP-OES) (Perkin Elmer, Shelton, USA).

Methods of plant analysis

Approximately 250 mg of ground, dried plant material was digested with 5 mL of concentrated H_2SO_4 and further oxidized with H_2O_2 (30% reagent grade) using the method of Thomas et al[24]. The digestate was thoroughly mixed with distilled water and brought to 100 mL. The solution was then allowed to stand overnight to allow the silica to settle. The amount of nitrogen in the solution was determined using the Kjeldahl method and titration; the amount of P in the solution was determined by a yellow phosphor–vanado–molybdate complex[25] and the metal ions were analyzed using ICP-OES (Perkin Elmer, Shelton, USA).

	Data			Day after planting (DAP)			Day after treatment (DAT)		
Activity	**Gondang**	**Tawar**	**Selangor**	**Gondang**	**Tawar**	**Selangor**	**Gondang**	**Tawar**	**Selangor**
Planting	26/10/2022	26/10/2022	26/10/2022	0	0	0			
1st cut	26/12/2022	28/12/2022	21/01/2022	61	63	87			
Liming Treatment NPK fertiliser	2/01/2023 12/01/2023	11/01/2023 11/01/2023	1/06/2023 1/06/2023	68 78	74 74	218 218	0 10	0 0	0 0
2nd cut	30/01/2023	10/02/2023	2/08/2023	96	104	280	28	30	62
NPK fertiliser	1/02/2023	14/02/2023	3/08/2023	98	108	281	30	34	63
3rd cut	15/03/2023	10/04/2023	27/10/2023	140	163	367	72	89	149
NPK fertiliser	17/03/2023	12/04/2023	27/10/2023	142	165	367	74	91	149
4th cut	22/05/2023	14/07/2023	8/01/2024	208	258	458	140	184	240
NPK fertiliser	22/05/2023	14/07/2023	8/01/2024	208	258	458	140	184	240
5th cut	2/08/2023	10/10/2023	25/06/2024	280	347	627	212	273	409

Table 1. Work scheduling during this study.

All the chemicals used in this study were of analytical grade and had a purity level of 99.9%, and were obtained from Sigma Aldrich Chemie GmbH (Munich, Germany).

Statistical analysis

All the data were analyzed using Statistical Analysis System (SAS) Version 9.4 software[26]. All the data were subjected to analysis of variance (ANOVA) to detect the variations in plant response at different sampling times. A comparison of means was performed using the least significant difference (LSD) test.

Results

Soil characteristics

The soils used in this study include the Gondang, Selangor and Tawar soil series. Their physicochemical characteristics are presented in Table 2. Soil pH ranges spanned from 4.0 for the Gondang soil to 4.7 for the Tawar soil (Table 2). Gondang soil had the highest electrical conductivity (EC) with a value of 3.90 dS/m, while EC was 0.03 and 0.07 dS/m, respectively, for the Tawar and Selangor soils. The highest value for organic C was (28.13%) for the Gondang soil and 2.02 and 6.20%, respectively for the Tawar and Selangor soils. The same trend was also observed for organic matter content and cation exchange capacity (CEC) of the soils. The CEC was highest for the Gondang soil (84.75 cmol (+)/kg soil) compared with 18.60 and 6.70 cmol (+)/kg soil for the Selangor and Tawar soil series, respectively. Presence of major elements, such as Na, K, Ca and Mg was low (< 1.0 cmol (+)/kg soil) in these soils. However, S content was highest (0.44%) for the Selangor soil as compared to 0.01 and 0.14%, for the Tawar and Gondang soils, respectively. Soil particle size analysis showed that the main soil components were silt (51.00%) for the Gondang soil and sand (79.90%) for the Tawar soil. Similar content of sand, silt and clay contents of about > 30.00% was observed for the Selangor soil. All other physicochemical characteristics are given in Table 2.

Water solubility test of POLY4

The main constituents of POLY4S and POLY4G were K, Ca, Mg, and S. Other constituents include Na and Cl (Table 3). Slightly higher content of K, Ca, Mg and S were observed in POLY4S as compared to POLY4G.

Evaluation of nutrients released in an open leaching soil column experiment

Elemental release pattern of POLY4S

A laboratory simulation study using the Tawar soil series in an open leaching system revealed that cations, such as Na_2O, K_2O, CaO and MgO (Fig. 4), and anions, SO_4^{2-} and Cl^- (Fig. 6v), were released into solution. It was

Soil parameters	Unit	Soil sampling location		
Local name (label)		Simpang Renggam (SPR)	Sedili (SDI)	Sembrong (SBR)
GPS location		01°46′20.5″N 103°19′	01°57′ 16,5″N	02°16′59.2″N
		47.71″E, 160 m ASL	104°06′ 00″E, 9 m	103°30′20.3″E; 42 m
PH (H_2O)		4.00	4.6	4.7
Lime requirements to reach pH 5.5-6 mg NaOH/kg so		3,776.00	3,472.00	800.00
Electrical conductivity	dS/m	3.90	0.07	0.03
Organic carbon	%	28.13	6.20	2.02
Organic matter	%	48.66	10.73	3.49
Cation exchange capacity	cmol(+)/kg soil	84.75	18.60	6.70
Calcium	cmol(+)/kg soil	0.83	1.77	0.41
Magnesium	cmol(+)/kg soil	1.09	1.08	0.25
Potassium	cmol(+)/kg soil	0.13	0.26	0.07
Sodium	cmol(+)/kg soil	0.35	0.12	0.10
Nitrogen	%	1.04	0.27	0.09
Sulphur	%	0.14	0.44	0.01
Available phosphorus	mg/kg soil		26.40	0.75
Fine earth fraction in the mineral matter:				
Clay	%	39.40	37.20	13.10
Silt	%	51.00	31.30	7.00
Sand	%	9.60	31.50	79.90
Texture class		Silty clay loam	Clay loam	Loamy sand
Soil water at field capacity	%	31.00	36.56	26.12
USDA Soil Taxonomy27		Dysic, isohyperthermic,Typic Haplohemist	A fine, mixed,isohyperthermic, Fluvaquentic Dystrudepts	A fine, mixed,isohyperthermic, Typic Paleudults
Soil series		Gondang	Selangor	Tawar

Table 2. Physicochemical characteristics of the soils used in this study.

Parameters	Unit	POLY4 types	
		Standarc	Granular
Calcium	% CaO	22.60	21.54
Potassium	% K_2O	17.76	15.96
Magnesium	% MgO	8.38	7.40
Sodium	% NaO	0.20	0.40
Sulphur	% S	21.15	20.60
Chloride	% Cl	2.66	5.62

Table 3. Water-soluble content of the selected elements in the POLY4S and POLY4G forms[28]. AOAC solubility test

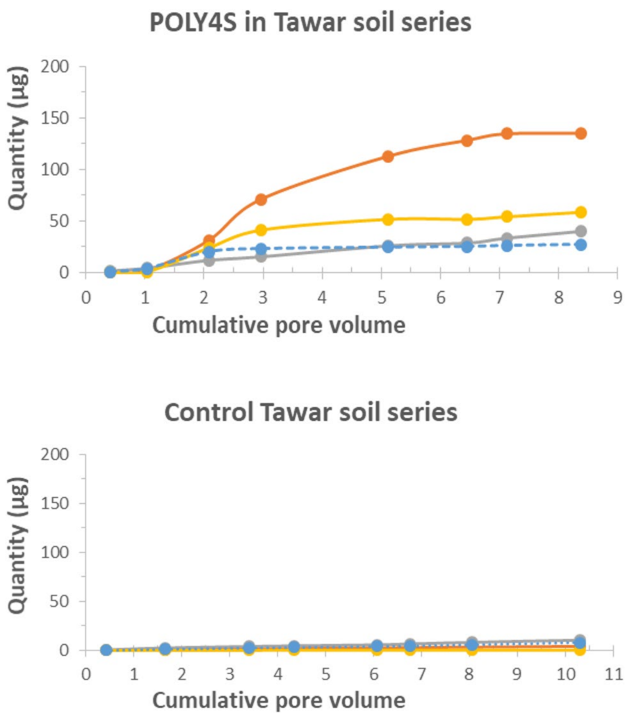

Fig. 4. Dissolution of POLY4S released cations, K_2O, MgO, CaO, and Na_2O, and anions, SO_4^{2-} and Cl^- (Fig. 6v), in solution.

observed that 27.14 µg Na_2O, 135.20 µg K_2O, 39.99 µg CaO and 58.51 µg MgO were released at the end of the leaching experiment, at PV of 8.89.

POLY4's main constituents are K_2O, CaO, MgO, SO_4^{2-} and other elements in small quantities[26]. These are very important macronutrients for plant growth and development. If the quantity of macronutrients is low and insufficient to support crop nutrient requirements, additional fertilizers, either alone or in combination with several elements, will still be needed.

Potassium release patterns for POLY4S in different soils

Since K_2O is a major macro-element required by most plants for growth and development, further investigation of POLY4S were conducted on soils with different characteristics. The concentration of K_2O released varied greatly between the soil types (Fig. 5i). In the Selangor soil series K_2O was released at higher concentrations earlier than in the Tawar and Gondang soils, reaching a maximum of approximately 160 mg K_2O/L, at 1.6 and 3.0 pore volume (PV) for the Selangor and Tawar soils, respectively. The Gondang soil showed the lowest release concentration of 32.95 mg K_2O/L at 7.9 PV.

Elemental release pattern from standard chemicals and fertilizers

The characteristics of a fertilizer, such as water solubility, are very important for nutrient supply and uptake by plants. Leaching of the sandy loam Tawar soil columns treated with K^+ from muriate of potash (60.0% K_2O; 47.0% Cl^-), Ca^{2+} from $CaCl_2$ $2H_2O$ (37.53% CaO; 48.3% Cl^-) and Mg^{2+} from $MgSO_4$ $7H_2O$ (16.4% MgO; 40.4% SO_4^{2-}) was compared with that treated with POLY4S. Variations in the concentrations of all nutrients, including SO_4^{2-} and Cl^- ions, were observed during leaching of the Tawar soil series (data not presented). However, the

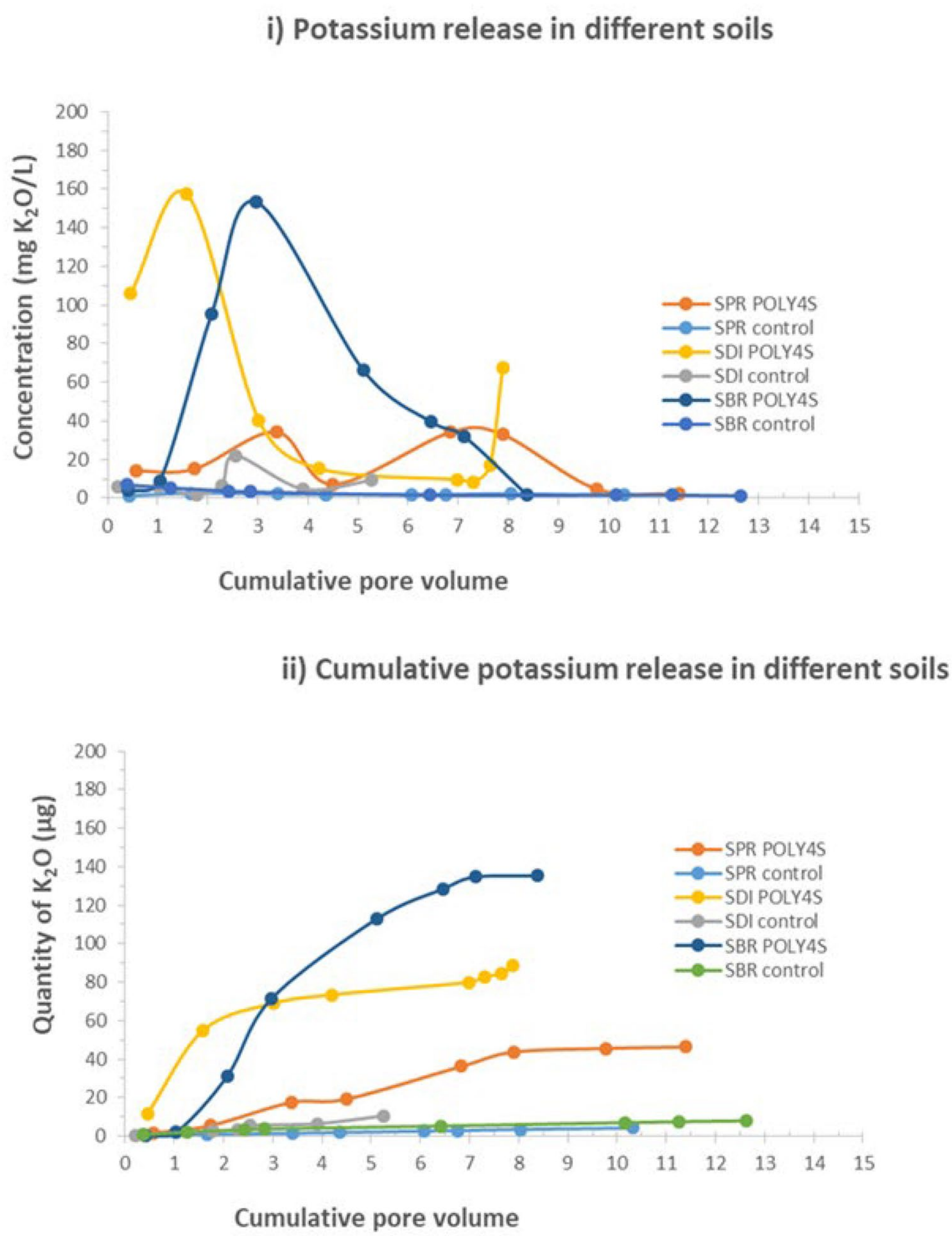

Fig. 5. Concentration of K_2O in the leachate (i) and its cumulative quantity (ii) with increasing PV of the Gondang, Selangor and Tawar soil series.

differences in trends of cation and anion release are clearly distinct when expressed in cumulative quantities against the advance of PV leaching (Fig. 6).

The release of Na_2O from POLY4S was rapid in the beginning compared to that from POLY4G. At the end of the experiment, Na_2O released reached approximately 27.0 µg at a PV of 8.4 for POLY4S and 17.9 µg at a PV of 7.9 for POLY4G (Fig. 6i). However, for the control, it increased gradually until the end, with a value of 13.7 µg at a PV of 12.6. Interestingly, K_2O release was very rapid at the beginning of the experiment for MOP (78.1 µg K_2O) at a PV < 2.0, and increased gradually until the end of the experiment (121.2 µg K_2O at 9.7 PV). The same trend was observed for POLY4S and POLY4G, when the PV was 5.0. However, at this PV range, the quantity of K_2O released was always greater from POLY4S than that from POLY4G (Fig. 6ii). However, by the end of the experiment both reached almost the same quantity (135.2 µg K_2O, PV 8.4 against 133.3 µg K_2O, PV 7.8) (Fig. 5ii). The control soil column presented the lowest amount of K_2O release throughout the entire experiment.

For CaO, at a PV < 2.0, the released quantity increased steadily for the soil columns treated with POLY4S and POLY4G. In contrast, the same trend was achieved only at a PV of 5.0 for $CaCl_2$ $2H_2O$ (Fig. 6iii). The quantity of CaO recovered at the end of the experiment was 40.0 µg for POLY4S, 28.5 µg for POLY4G, 27.6 µg for $CaCl_2$ $2H_2O$ and 14.7 µg for the control. The cumulative quantity of MgO increased rapidly at PV < 2.0 for all the fertilizers tested. With the exception of POLY4S, both POLY4G and $MgSO_4$ $7H_2O$ presented almost constant

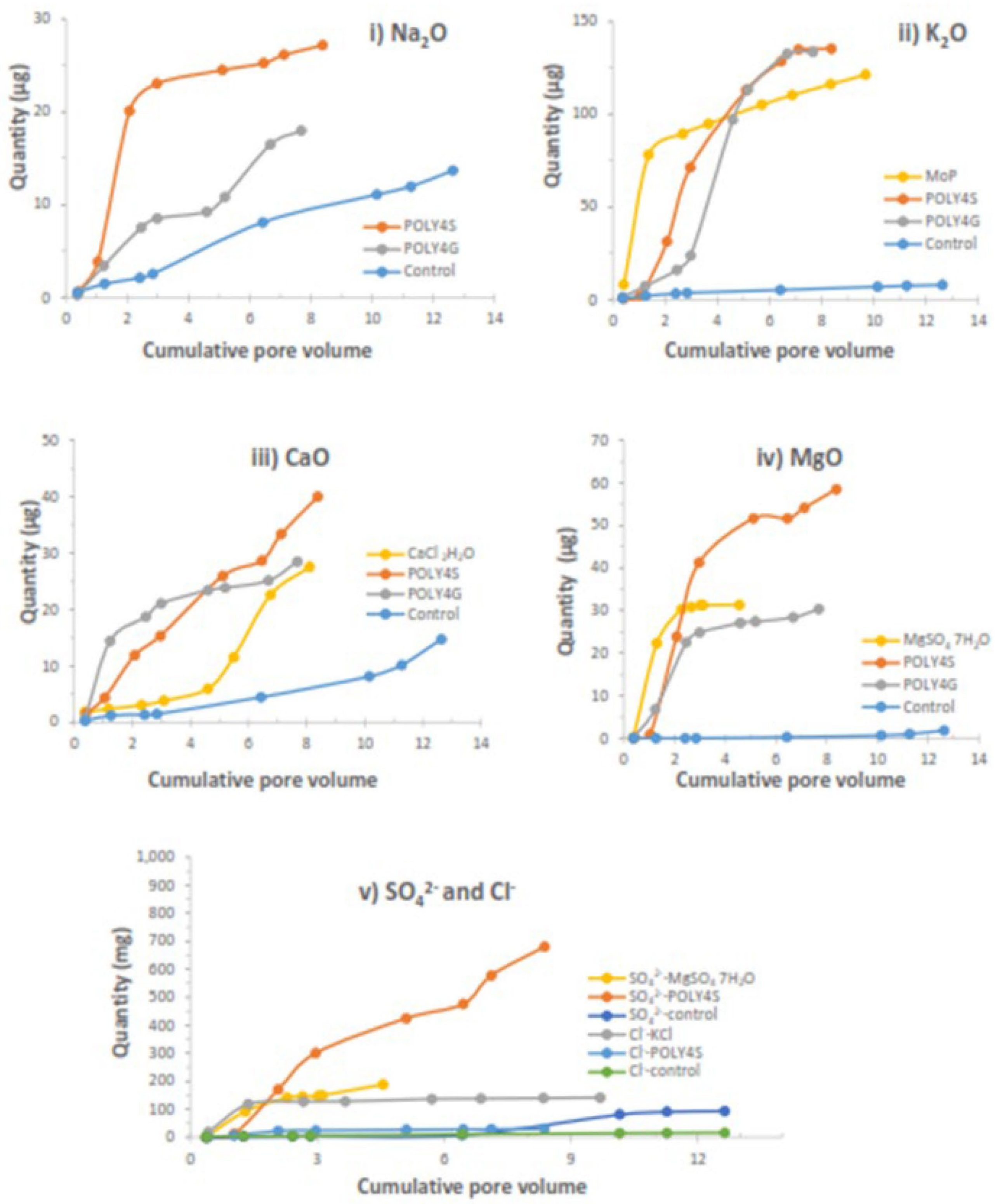

Fig. 6. Cumulative release of (i) Na_2O, (ii) K_2O, (iii) CaO, (iv) MgO and (v) SO_4^{2-} and Cl^- during leaching of the Tawar soil column relative to the most common ions in POLY4 compared with the standard chemicals. *Note* the control is the soil only column.

quantities of MgO up to the end of the experiment, with values of 30.3 µg at a PV of 7.7 (POLY4G) and 31.3 µg at a PV of 5.6 ($MgSO_4$ $7H_2O$). In contrast, the cumulative quantity for POLY4S was very high at 58.5 µg at a PV of 8.4. For the control soil column, the values were low throughout the study period, with an end value of 1.8 µg of MgO at a PV of 12.6 (Fig. 6iv).

Since POLY4 contains SO_4^{2-} in its structural formula and was mined under the seabed (Cl^- contamination), it was necessary to examine these two anions in this study. The cumulative quantity of both anions was lower (< 100 mg) at PV < 2, but the quantity of SO_4^{2-} released increased rapidly for POLY4S up till the end of the experiment, with a value of 679.9 mg at a PV of 8.4. In contrast, during the same period, the cumulative Cl^- ion content remained almost constant and reached 93.4 µg at a PV of 12.67. The highest cumulative quantity of SO_4^{2-} measured in the $MgSO_4$ $7H_2O$-treated soil column was 188.2 at a PV of 4.6. Further, the cumulative quantity of Cl^- from KCl was even greater than that from POLY4S (191.4 mg at a PV of 9.7) (Fig. 6v).

Residual nutrients in the soil column at the end of the leaching study

At the end of the leaching period of the Tawar soil column the concentration of residual Na_2O, K_2O, CaO and MgO displayed an almost similar trend with increasing soil column depth for the control, soil column treated with POLY4S, POLY4G and standard laboratory chemicals (Fig. 7). Mobility of CaO decreased up to 15–20 cm depth and increased to the highest value in the region of 20–25 cm soil depth.

Simulation of nutrient release from fertilizers and crop uptake in a controlled environment

Dry matter yield of *Napier* grass

The time taken to obtain a satisfactory weight size sample after treatment application differed greatly between the three tested soils. For example, for the first harvest, the duration was 28 days for the Gondang soil, 30 days for the Tawar soil and 62 days for the Selangor soil. Overall, the growth of the *Napier* grass in this study was monitored for 212 days for the Gondang soil, 273 days for the Tawar soil and 409 days for the Selangor soil, covering four harvests (Table 1).

The evolution of the *Napier* grass dry weight throughout this study, as indicated by the cumulative dry weight after the fertilizer treatments, differed greatly for each soil type (Fig. 8). However, at the end of the experiment, there was no significant difference in the ANOVA of the cumulative dry weight of *Napier* grass for the Gondang (Pr > F, 0.1362) and Tawar soil series (Pr > F, 0.1082) (data not presented). In contrast, there were highly significant differences between treatments (Table 4) for the Selangor soil series. For the Selangor soil series the highest cumulative dry weight of *Napier* grass was obtained from soil treated with POLY4 standard (284.7 g/polybag), followed by POLY4 granular (103.44 g/polybag) and the least with muriate of potash (92.58 g/polybag (Table 4). Overall, the average cumulative dry weight for POLY4 was greater than that for MOP, with values of 6.5% for Gondang, 109.6% for Selangor and 37.8% for Tawar soil series.

Evolution of selected nutrients' uptake in harvested *Napier* grass

The uptake of selected nutrients was calculated by multiplying the concentrations of the elements and the dry matter yield measured at each harvest, following the addition of MOP, POLY4G and POLY4S to the soils.

The trend of cumulative nutrient uptake increased with the advance of harvesting time and varied greatly between the different fertilizer treatments in the Gondang soil series (Fig. 9). The same pattern was also observed for the cumulative nutrient uptake for the Tawar (Fig. 10) and Selangor (Fig. 11) soil series.

At the end of the experiment the ANOVA table showed that there were no significant differences in the cumulative uptake of Na_2O (Pr > F, 0.1952), K_2O (Pr > F, 0.2177) and MgO (Pr > F, 0.1676); but there were significant differences for CaO (Pr > F, 0.0128) uptake, in the Gondang soil treated with the different fertilizers. The same pattern was also observed in the Tawar soil series, where Na_2O (Pr > F, 0.6612), K_2O (Pr > F, 0.0672) and MgO (Pr > F, 0.1659) uptake did not show significant differences, while CaO (Pr > F, 0.0128) uptake showed significant differences between treatments. In contrast, for the same fertilizer treatments, the Selangor soil series showed significant differences in cumulative K_2O (Pr > F, 0.0068), CaO (Pr > F, 0.0052) and MgO (Pr > F, 0.0066) uptakes, but not for Na_2O (Pr > F, 0.0672) uptake. The cumulative uptake of selected nutrients at the end of experiment is shown in Table 5.

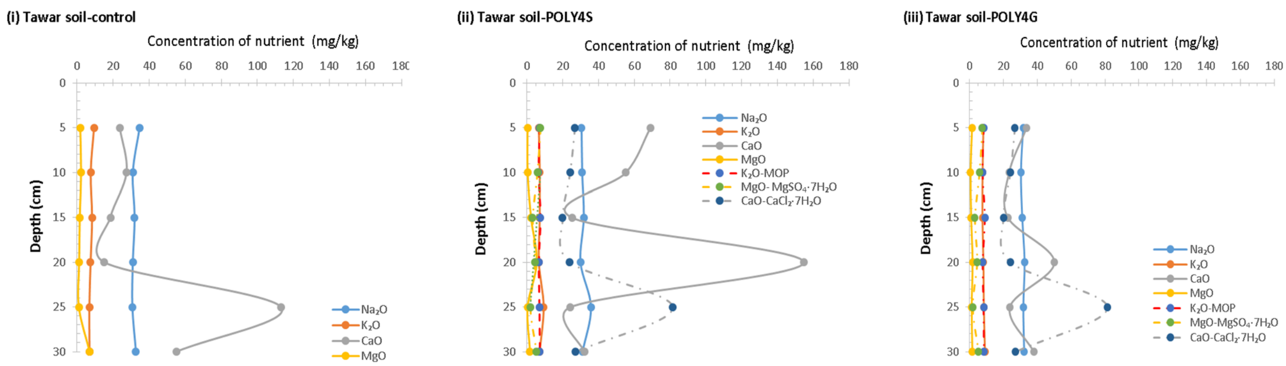

Fig. 7. Residual Na_2O, K_2O, CaO and MgO in the Tawar soil column: (i) Non-treated (control), (ii) treated with POLY4S and (iii) treated with POLY4G in comparison with MOP, $MgSO_4.7H_2O$ and $CaCl_2.2H_2O$ for respective elements at the end of the experiment.

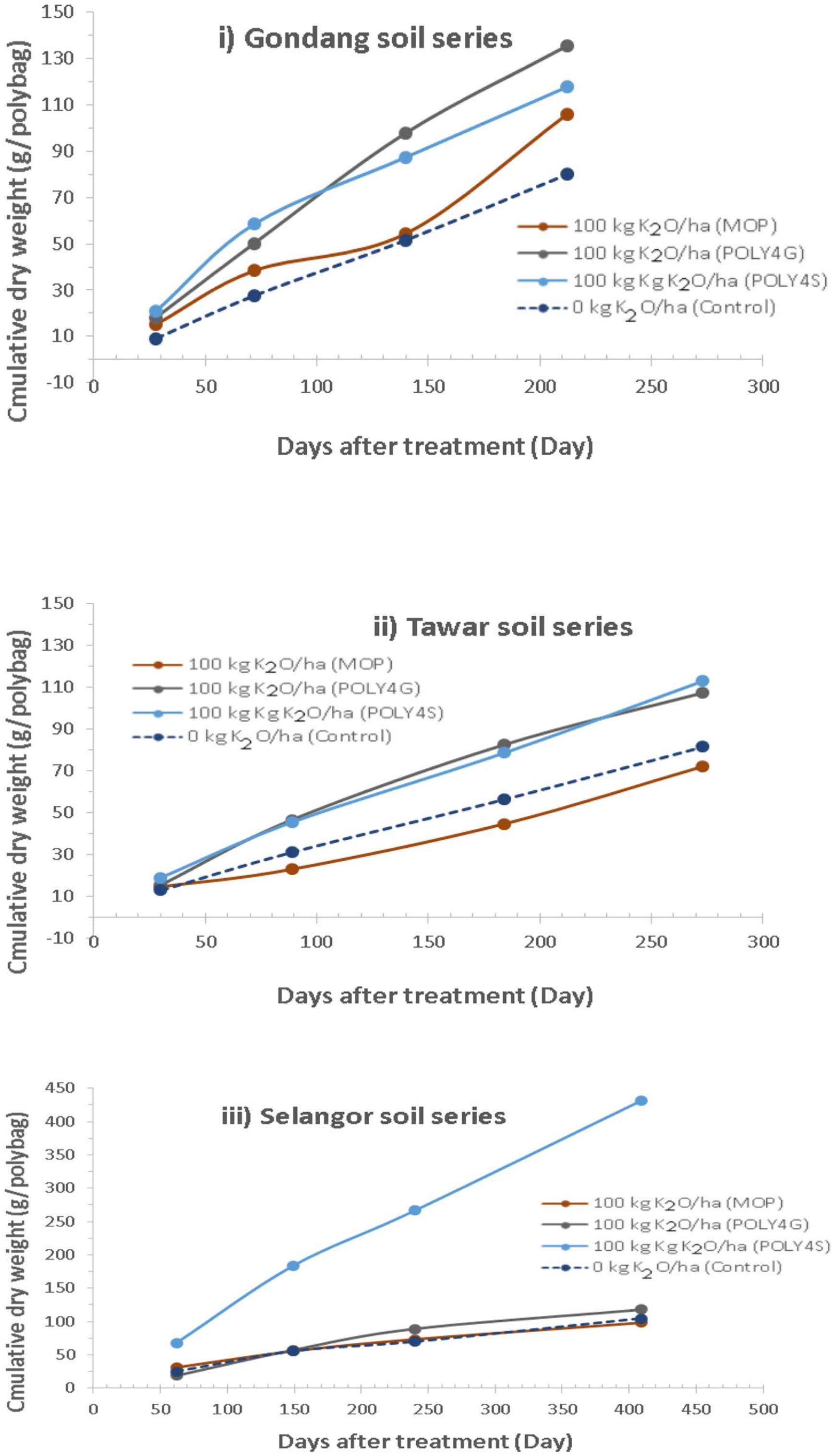

Fig. 8. Cumulative dry weight of *Napier* grass after fertilizer treatment application for: (i) Gondang-, (ii) Tawar- and (iii) Selangor-soil series.

Soil characteristics at the end of the experiment

The soil pH ranged from 3.7–3.8 for the Gondang, 4.9–5.3 for Tawar, and 3.5–3.6 for the Selangor soil series. Meanwhile, the average, EC (µS/cm) ranged from 575 to 622 for the Gondang, 78 to 90 for the Tawar, and 637 to 843 for the Selangor soil series. The N and P contents were almost consistent across all the soil types, from 4.7 to 5.4% for N and 107.8 to 191.0 mg for P_2O_5/kg soil. Chloride content was consistently low, 0.02 to 1.1% for

Soil series	Cumulative dry weight of Napier grass (g/polybag)			LSD value at 5% level, N = 6
	POLY4			
	Standard	Granular	Muriate of potash	
Gondang	91.63	112.20	95.72	37.55
Selangor	284.7 a	103.44 b	92.58 b	111.61
Tawar	96.99	100.33	71.58	25.65

Table 4. Cumulative dry matter yield of *Napier* grass following the addition of POLY4 in standard and granular forms and muriate of potash to the three different soil series. The same letter in a row is not significantly different at 5% level.

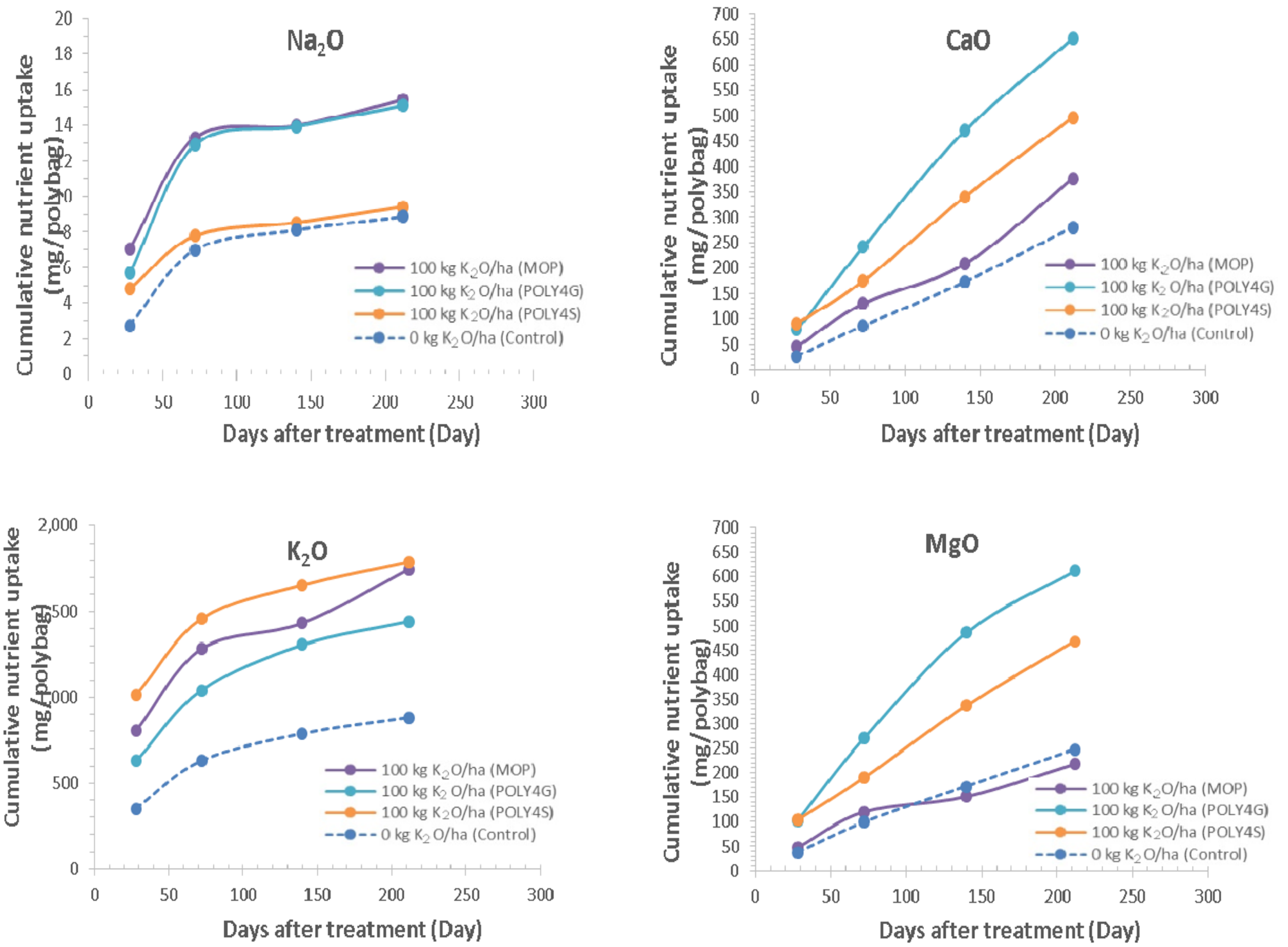

Fig. 9. Cumulative nutrient uptake of Na_2O, K_2O, CaO and MgO by *Napier* grass after fertilizer treatment application for the Gondang soil series.

all the soil types after the different fertilizer treatments. The concentrations of other elements, such as Na, K, Ca and Mg, were consistent across all soil types and treatments in this study (Supplementary data available—II).

Discussion

The Gondang soil series contained 48.7% organic matter, which classifies it as organic soil. In contrast, organic matter content was low in the other soil types, Selangor (10.7%) and Tawar (3.5%). The Selangor soil series was developed from marine clay materials containing some clay loam mottling in the topsoil profile (< 20 cm soil depth). This soil series was influenced by saltwater during sea tides. Compared with the Gondang and Tawar soils, the Selangor soil series was imperfectly drained and had the highest water content at field capacity (36.56%). As expected, the S content in this soil was greater (0.44%) than that in the Gondang and Tawar soil series (< 0.1%) because of sulfate accumulation in the soil during sea tides. The Tawar soil series developed on sub recent alluvium and contained mainly coarse sandy materials, which were perfectly well drained. Analysis

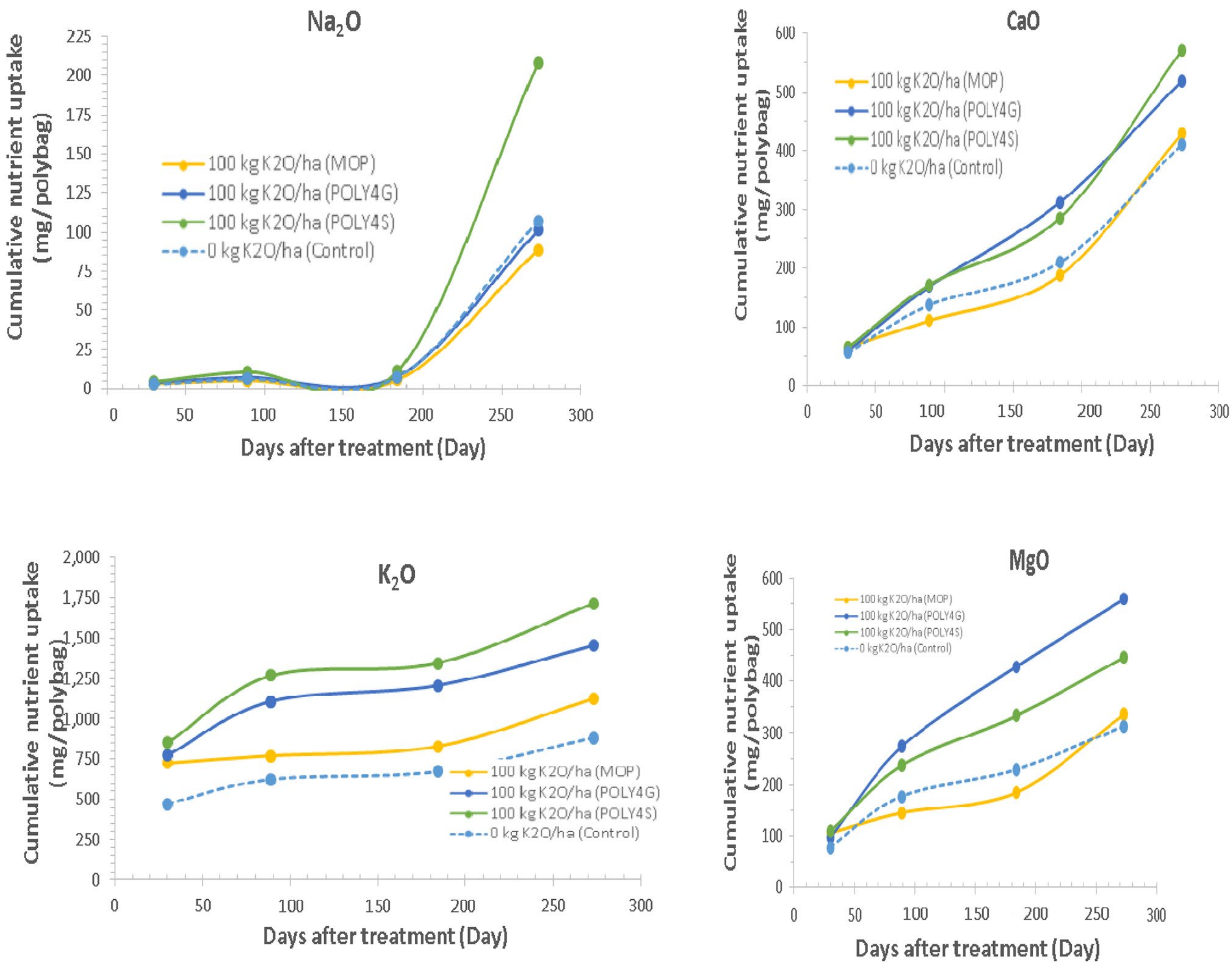

Fig. 10. Cumulative nutrient uptake, Na_2O, K_2O, CaO and MgO by *Napier* grass after the application of fertilizer treatments for Tawar soil series.

of nutrient availability (Table 2) showed that the Gondang soil series had higher nutrient content than that in the Selangor and Tawar soil series. It is therefore natural for the Gondang soil series to have better fertility status than the other two soil series.

When POLY4 is applied to soil, its dissolution depends not only on its own characteristics, but also on the soil properties, crop types and environmental factors. The ions released may be absorbed by soil particles or organic materials, such as humic acid components, and/or absorbed by plants through root systems and transported to other plant parts, such as leaves and fruits. Ion that are not adsorbed are subject to leaching loss through the soil profile to underground water, rivers and finally to the sea. The mode(s) of ion release and ion movement in the soil, water and plant ecosystems are therefore very important aspects in the assessment of POLY4 for agricultural use.

Both POLY4S and POLY4G completely dissolved in water. The dissolution of POLY4 released major nutrients, Ca, K, Mg and S, into the solution. The values of the selected elements soluble in water for POLY4S and POLY4G are presented in Table 3. In general, all the selected elements showed a slightly higher value in POLY4S as compared to POLY4G. The main elements from POLY4S were K (17.76% K_2O), Ca (22.60% CaO), Mg (8.38% MgO) and S (21.15% S). The values obtained for the selected elements in this study (Table 3) displayed the same trend as that reported by the Bureau Veritas, Switzerland[29] (Supplementary data available – I). At the same time some other minor components were also released, attributable to contamination during POLY4 mining under the seabed. These impurities may have contributed to the values of Na_2O (0.2 to 0.4%) and Cl^- (2.7 to 5.6%) recorded for both POLY4 standard and granular types, respectively (Table 3).

Based on the results of POLY4S leaching in Tawar soil, the following trend of nutrient release was observed: $K_2O > MgO > CaO > Na_2O$. However, this trend differs from the results obtained from total analysis of POLY4, in which the contents of the respective nutrients were 14.0, 6.0, 17.0 and 1.6%, respectively. This could be because although the POLY4 content of CaO was greater than that of MgO and K_2O, its dissolution in the soil column was likely restricted by the structure of the polyhalite minerals or adsorbed by soil colloids. The residual concentration of CaO in the soil column at the end of the leaching experiment confirmed a higher

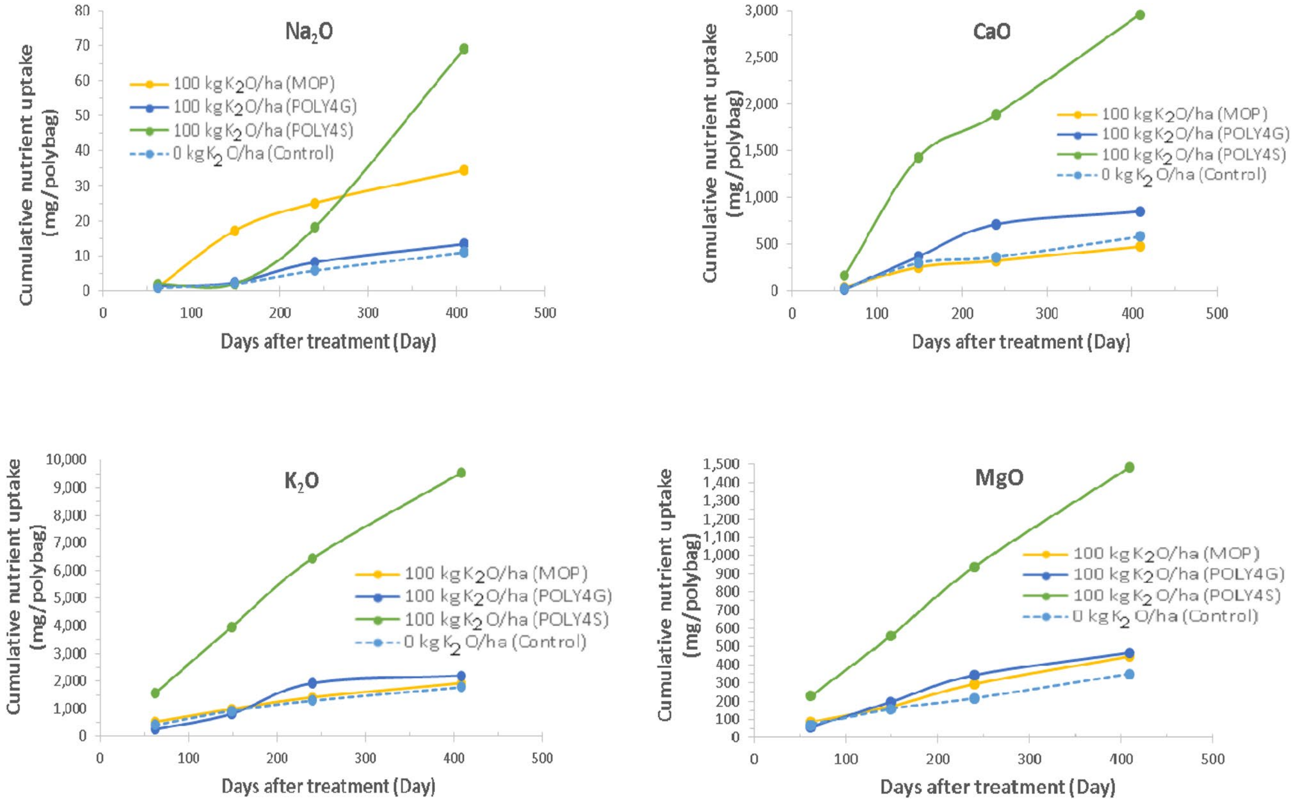

Fig. 11. Cumulative nutrient uptake, Na_2O, K_2O, CaO and MgO by *Napier* grass after the application of fertilizer treatments for Selangor soil series.

accumulation of Ca^{2+} in the column at a depth of 20–25 cm for Tawar soil treated with either POLY4S, POLY4G or $CaSO_4.2H_2O$ (Fig. 7). The average concentrations of CaO in these columns were 60.12 mg/kg soil for the POLY4S treatment, 31.92 mg/kg soil for the POLY4G treatment and 33.90 mg/kg soil for the $CaSO_4.2H_2O$ treatment. In contrast, the values for K_2O and MgO were low. The average concentration of K_2O ranged from 7.36 mg/kg soil for POLY4S to 8.34 mg/kg soil for MOP, whereas, for MgO, it ranged from 1.29 mg/kg soil for POLY4G to 4.67 mg/kg soil for $MgSO_4.7H_2O$. In the polyhalite crystal structure, the calcium ions (Ca^{2+}) have a coordination number of eight, where each Ca^{2+} is bonded to eight O atoms, forming an eight-vertex polyhedron (specifically described as a triangular dodecahedron) within the crystal lattice. This configuration makes the Ca^{2+} a very stable ion, resulting from the loss of its two 4 s valence electrons[30]. In this type of polyhalite mineral structure, the Mg component is highly soluble, while in contrast the Ca component is sparingly soluble, when exposed to water. Furthermore, the low solubility of the Ca^{2+} ion can also be attributed to the formation of the mineral gypsum ($CaSO_4.2H_2O$), which has limited water solubility (around 2.03 g/L), whereby gypsum is precipitated during polyhalite dissolution[13]. This low solubility is a key characteristic of the Ca^{2+} ion, often leading to constant residual Ca concentrations in solution once saturation limit (K_{sp}, solublity product constant) is reached. Hence, the availability of CaO to plants is severely limited.

The concentration of all elements measured in this study subsequently decreased tremendously with increasing leaching volume. Although K_2O was released faster in the Selangor soil series than in the Tawar soil series, the overall quantity of K_2O release exhibited different trends when cumulative amount released was computed (Fig. 5ii). These results suggest that soil characteristics play an important role in the release of K_2O into the soil solution, where the Tawar soil series consists of loamy sand, Selangor soil series consists of clay loam and Gondang soil series consists of peat and sandy clay loam. Among the three soils, the Tawar soil series had the lowest cation exchange capacity (CEC) (6.7 cmol (+)/kg soil), Selangor recorded higher capacity (18.6 cmol + / kg soil) and Gondang had the highest (84.75 cmol (+)/kg soil). This is pertinent as although the K_2O from POLY4S is generally water soluble it is retained by the soil particle charge, which limits its availability to plants.

The release of common cations and anions from POLY4 and the standard chemical fertilizers during leaching of the Tawar soil series showed different patterns (Fig. 6). These results suggest that under normal conditions of fertilizer use, the solubility and hence, availability of POLY4S elements were more pronounced than that of POLY4G and the other chemical fertilizers. In the case of cumulative MgO, its release from POLY4S was more pronounced as compared to the other Mg sources, and thus was comparatively more readily available. Further, the relative amount of SO_4^{2-} content in POLY4S was approximately 3.6-fold greater than that in $MgSO_4$ $7H_2O$, whereas the Cl^- content was approximately 0.5-fold lower than that in KCl. This result is in agreement with other published results that state that POLY4 contains high amounts of SO_4^{2-} and trace quantities of Cl^-. The presence of Cl^- in POLY4S is likely due to contamination during mining of POLY4 from under the seabed.

Fertilisers	Element in oxide form	Soil series		
		Gondang	Selangor	Tawar
		mg/polybag		
MOP	Calcium	297.68b	408.80b	348.08b
POLY4G		450.93a	583.70b	489.16a
POLY4S		341.01b	1790.00a	430.42b
ANOVA table		S	S	S
LSD value at $p = 0.05$		142.5	806.26	99.18
MOP	Potassium	1,318.40	1652.00b	862.20
POLY4G		1,131.30	1709.00b	1401.20
POLY4S		1,259.90	6155.00a	1211.42
ANOVA table		NS	S	NS
LSD value at $p = 0.05$		794.73	2918.40	570.28
MOP	Magnesium	236.40	378.80b	295.35
POLY4G		449.20	390.50b	507.95
POLY4S		334.10	951.50a	337.15
ANOVA table		NS	S	NS
LSD value at $p = 0.05$		259.55	412.73	202.83
MOP	Sodium	12.52	21.42	93.34
POLY4G		11.45	9.86	118.06
POLY4S		9.32	43.74	148.58
ANOVA table		NS	NS	NS
LSD value at $p = 0.05$		5.42	36.53	124.81

Table 5. Average cumulative (N = 6) amount of selected elements taken up by *Napier* grass grown in the Gondang Selangor and Tawar soil series at the end of the study. Cumulative nutrients uptake represented by 4-harvest after DAT for Gondang, Selangor and Tawar soil series, respectively; NS (Pr>F >0.05); S (Pr>F <0.05); Means with the same letter in the column are not significantly different.

In this study, only the cumulative dry weight of *Napier* grass grown on the Selangor soil series showed a significant difference (Table 4) in comparison to that grown in the Gondang and Tawar soil series. This could be related to the time taken for early establishment of the *Napier* grass (Table 1) due to the texture of the respective soils. Though the *Napier* grass took a very long time to establish in the clayey soil textured Selangor soil series, but in the long term the grass would be able to absorb the nutrients from the fertilizers added to the soil. Additionally, with proper water management, the soil moisture content can be maintained throughout the growing period.

The element release efficiency of POLY4 can be estimated from the results provided by elemental analysis[26], based on the data in Table 5. Theoretically, for every unit of K released by POLY4, congruently 1.03 units of CaO and 0.35 units of MgO are dissolved and made available to plants. However, in this study, on average, the above values were 0.33 for CaO and 0.28 for MgO. This suggests that although POLY4 contains high amounts of total Ca (13.0% CaO) and a lower amount of Mg (4.39% MgO), only a small proportion of Ca^{2+} and greater amounts of Mg^{2+} were taken up by the *Napier* grass (Table 5), relative to the K content in POLY4 (Table 6). In terms of nutrient recovery from the total available amount, MgO recovery was greater at 80.8%, whereas CaO recovery was only 32.5%, based on average POLY4S and POLY4G results across all soils used in this study. This finding is consistent with the trend of nutrient concentrations during each harvest, in which the CaO concentration decreased gradually during the first 3 harvests (at approximately 150 DAT) and K_2O and MgO decreased very rapidly during the same sampling periods. This trend is also in agreement with the results from the open leaching of the Tawar soil column for a period of 30 d (Fig. 5iii and 5iv), where the quantity of MgO released was higher than that of CaO. The reason for this, as explained earlier, is due to the chemical structure of mineral polyhalite and the incongruent dissolution of Ca^{2+} and Mg^{2+}, which results in precipitation of Ca^{2+} as gypsum, which has low water solubility. These results suggest that in any fertilizer formulation where POLY4 is used as a source, the crop requirement for Mg should be taken into consideration. There were no comparison can made based on the dynamic of nutrients release from POLY 4 under acid tropical conditions, except on deserts soil by Yermiyahu et al[13] and on red lithic sandy soil by Barbarick[12]. The present study is restricted to a controlled condition in the laboratory using an open leaching system and in shade house experiment of limited soil volume. However, under natural filed condition the factors of soil, crop types and environmental condition could change the results.

Conclusions

POLY4G and POLY4S are completely soluble in water, and release Ca, K, Mg, and SO_4^{2} ions, with some impurities, such as Na^+ and Cl^- ions, that are present in negligible quantities. When POLY4 is dissolved in an open leaching soil column, the release of cations depends on the soil characteristics, such as the cation exchange capacity (CEC) and soil texture. Compared with the Gondang soil (PV 11.4; 46.3 μg K_2O), the loamy clay

Fertilisers	Element in oxide form		Elemental ratio to K in MoP			Element (%) in POLY4	Ratio to K	Element released (%)
			Gondang	Selangor	Tawar			
MOP	Calcium		0.23	0.25	0.40	13.00	1.03	32.5
POLY4G			0.40	0.34	0.35			
POLY4S			0.27	0.29	0.36			
		Average for POLY4	0.33	0.32	0.35			
		Average across soil		0.33				
MOP	Potassium							
POLY4G						12.62	1.00	
POLY4S								
MOP	Magnesium		0.18	0.23	0.34	4.39	0.35	80.8
POLY4G			0.40	0.23	0.36			
POLY4S		Average for POLY4	0.27	0.15	0.28			
			0.33	0.19	0.32			
		Average across soil		0.28				
MOP	Sodium		0.01	0.01	0.11			
POLY4G			0.01	0.01	0.08			
POLY4S		Average for POLY4	0.01	0.01	0.12			
			0.01	0.01	0.10			

Table 6. Theoretical relative atomic elements in POLY4 for every unit of its K release compared with the cumulative uptake by *Napier* grass

textured Tawar soil released much greater quantities of K (PV 8.4; 135.2 µg K_2O) with high amounts of hemist materials. However, the subsequent uptake of this ion can be modified by plant roots.

With the exception of the Selangor soil series, cumulative dry matter yield of *Napier* grass did not significantly differ among the MOP, POLY4G and POLY4S treatments. However, POLY4 consistently increased dry matter yields in all the tested soil series. This is consistent with the results obtained in the leaching study for the three soils.

Further analysis of the average nutrient uptake for all harvests showed that for every unit of K released by POLY4, Ca uptake by the *Napier* grass was much lower than that of Mg, even though POLY4 contained more Ca (13.0% CaO) than Mg (4.39% MgO). Further, the nutrient recovery by *Napier* grass was greater for Mg (80.8% as MgO) than for Ca (32.5% as CaO) for every unit of K released. Hence, any future fertilizer formulations using POLY4 as the source needs to take Mg requirements into consideration.

Data availability

Any raw data may be request through corresponding author at mmhanafi@upm.edu.my

Received: 7 August 2025; Accepted: 28 February 2026
Published online: 09 March 2026

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Acknowledgements
We wish to express want to express our gratitude to Universiti Putra Malaysia (UPM), Crop Nutrients Division, Anglo-America PLc. and UPM Consultancy Services Sdn. Bhd (UPMCS). We are fully acknowledged the staff members of the Institute of Tropical Agriculture and Food Security (ITAFoS), UPM, the Department of Land Management, Faculty of Agriculture, UPM, Serdang, Selangor, Faculty of Science and Technology, Universiti Kebangsaan Malaysia (UKM) for supporting in the field, laboratory data collection and technical services during this study.

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Conceptual, design, data collection and analysis, draft and finalized the manuscript, M.M.H; management of fund and design, D.S; management of the funding, R.M., R.M and A.S.

Funding
This research received a partial external funding.

Declarations

Competing interests
The authors declare no conflict of interest and agreed for publication of this manuscript.

Additional information
Supplementary Information The online version contains supplementary material available at https://doi.org/10.1038/s41598-026-42984-0.

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