

Estimation of nutrient values of pig slurries in Southeast Spain using easily determined properties

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Abstract

The contents of available nutrients in pig slurries are not easy to quantify in situ without laboratory facilities, but chemical analyses using standard laboratory methods also take time and are costly and not practical for most farms. Thus, when animal slurries are applied to land, their fertiliser potential is often unknown. In addition, in the last years, the changes in the management of industrial piggeries has changed the nature of the pig slurries *vg.* decrease of the dry matter content, and consequently the methods and equations used for estimating the nutrient contents in these residues must be checked. In our study, slurry samples were collected from the storage tanks of 36 commercial farms in Southeast Spain. Samples were analysed for pH, electrical conductivity (EC), redox potential (RP), specific density (D), total solids (TS), sedimentable solids (SS), biological oxygen demand (BOD_5), chemical oxygen demand (COD), total nitrogen (TKN), ammonium nitrogen (AN), organic nitrogen (ON), and total contents of phosphorus, potassium, calcium and magnesium. Relationships between major nutrient levels of pig slurries and a range of physical and chemical properties were investigated. We also analysed the variability of pig slurries according to the production stage. TKN, AN and K were closely related to EC. The P content in slurries was related more closely to solids-derived parameters such as D. The use of multiple properties to estimate nutrient contents in pig slurries, especially for AN and K, seemed unnecessary due to the limited improvement achieved with an additional property. Therefore, electrical conductivity seemed to be the most appropriate single, easily determined parameter for estimation of total and ammonium nitrogen and potassium in pig slurries, with more than 83% of the variance explained. P seemed to be the worst key nutrient for estimation using any easily determined parameter.

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1. Introduction

Animal manure application to soils in arid and semi-arid areas was a traditional source of nutrients and organic matter for soil–plant systems (Villar *et al.*, 2004) but the scarcity of these biosolids in the second part of the last century reduced their use in the Southeast of Spain. But now, the increase in the number of industrial farms, without soil nearby, represents a new opportunity to reuse these materials for agricultural purposes, as a source of nutrients and organic matter. The recent manure production within Spain has been approximately

190×10^6 t year⁻¹ (Water Research Centre, 2001). The agricultural use of these manures is recommended, not only for fertilising but also to facilitate the disposal of these increasingly important residues.

Piggeries are one of the most industrialised farm activities, therefore representing a problem with respect to the management of the high volume of waste materials produced. Agricultural slurries contain useful amounts of several macronutrients. A sustainable use of pig slurries for fertilising purposes must start with a complete characterisation, to avoid rapid release of nutrients and to reduce negative environmental impacts, such as contamination of subsurface water due to leaching of nitrate (Vervoort *et al.*, 1998). However, the complex nature of these residues usually induces a very

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heterogeneous analytical composition, with a high space and time variability. Additionally, chemical analyses using standard laboratory methods are accurate but take time and are costly and not practical for most farms. Thus, when agricultural slurries are applied to land, their fertilizer potential is often unknown. Hence, the spreading procedure becomes one of waste disposal rather than nutrient utilisation, therefore increasing the risks of air, water and soil pollution (European Environment Agency, 2000). Knowledge of a range of properties might provide useful practical estimates of plant nutrient concentrations in slurries. However, in the last years, the changes in the management of industrial piggeries has changed the nature of the pig slurries *vg.* decrease of the dry matter content, and consequently the methods used for estimating the nutrient contents in these residues must be checked and updated. In accordance to this, Tunney (1978) studied the relationships between dry matter, specific gravity and nutrient contents of cattle and pig slurry, found a range of dry matter values for pig slurries between 5% and 25%. Now, the average value in Spain is around 1–5%.

Special attention must be paid to the possible correlations between certain easily determined parameters like pH, electrical conductivity, density, redox potential and dry matter and nutrients in pig slurries, in order to estimate the fertiliser application to agricultural soils (Scotford et al., 1998a).

Piccinini and Bortone (1991) found a high linear correlation between total Kjeldahl nitrogen and total solids in Italian pig slurries. Scotford et al. (1998b) observed that phosphorus was most closely related to two simple parameters, density and pH, in a multivariate analysis for agricultural slurries (cattle and pig) in the United Kingdom, Ireland, Germany and Italy. Van Kessel et al. (1999) found that density determination is one of the simplest methods available and the only one that can predict both total nitrogen and total phosphorus.

The aims of this experiment were to: (i) evaluate the nutrient content, especially N, P and K, of the pig slurries in Southeast Spain, (ii) analyse the variability of pig slurries according to the production stage, and (iii) find relationships which would make it possible to estimate NPK nutrient contents of these wastes from easily determined parameters.

2. Materials and methods

2.1. Description of the selected farms and sample collection

In the studied area (Vega Baja, in the Valencia State of Spain), piggeries are an increasing activity. In this area, over 120 pig farms were identified, and 36 were selected in order to obtain representative sam-

ples to account for potential variation in livestock management techniques. The monitored farms represent more than 75% of the pig production in the studied area, with an estimated total annual production of pig slurries of over 70,000 m³. The management types in these farms and the relative pig slurry productions (% in brackets) were: completed cycle, 43% (49%); nursery, 16% (17%); semi-completed cycle without fattening, 32% (28%); just weaners, 9% (6%). The relative pig slurry production as a function of production stage in the studied farms was: finishers (34%) > gestating sows (29%) > weaners (18%) ≈ farrowing sows (18%). In the majority (95%) of the farms, the management system is based on dry-feeding with pellets and/or flour. The pig slurries were collected mainly below the animal, in separate pits using a fully slatted floor system, and stored in swine lagoons or directly applied to soils.

In each farm, pig slurries were sampled separately from the pits according to the production cycle (gestating sows, farrowing sows, weaners, finishers). In order to minimize the variability between farms, the pig slurry must be stored in the pit between 30 and 60 days before sampling. After the mechanical homogenisation of each pit, a 25-L sample was obtained by integration of three 10-L subsamples and immediately analysed in the laboratory.

2.2. Analytical methods

pH, EC and Eh were determined potentiometrically in the raw or diluted pig slurries according to APHA (1998). D was measured by a hydrometer reading taken 15 s after stirring (Chescheir et al., 1985). TS were determined gravimetrically, after drying in a forced-aerated oven at 105 °C. SS were measured by natural sedimentation, in an Imhoff vessel, after 60 min (APHA, 1998). BOD₅ and COD were determined according to APHA (1998). TKN was determined by the Kjeldahl method. AN was determined also by steam distillation (APHA, 1998). Phosphorus was determined, after acid digestion, colorimetrically as the phosphomolybdate complex after reduction with ascorbic acid (APHA, 1998; Zhu et al., 2003). Total potassium, calcium and magnesium were determined, in the above digestion, by atomic absorption spectrophotometry.

2.3. Data analysis

Standard deviation and ANOVA for each analytical measure were performed in each production stage. In addition, the Combined Pig Slurry concept (CPS) was established as an estimation of the “typical” pig slurry composition produced in each farm considering the different analytical nature of pig slurry, depending on each production stage, and weighting by the relative flow rate

of every stage according to the Spanish and Valencia state guidelines (BOE, 2000; DOGV, 2000).

Pearson correlations and single and multiple regression analyses were carried out, in order to find simple equations to estimate the TKN, AN, P and K composition of pig slurries from easily determined parameters, using the SPSS 11.5v programme. The following generalised equation was used in linear regression analyses:

$$Y = wC_i + xC_j + z,$$

where Y = TKN, AN, P or K (kg m^{-3}); w , x and z are regression coefficients (x coefficient equal to zero in single property regression); and C_i and C_j are the measured properties. Percentages of explained variance (PEV) were used to measure the overall effectiveness of the linear regressions.

3. Results and discussion

3.1. Chemical analysis

The average pH in CPS was 7.45, with a wide variation, mainly due to the low pH value of the weaners (see Table 1). The pH of the water usually used in the studied farms ranged from 7.5 to 7.9, due to the high contents of calcium and magnesium. The pH of the pig slurries favours ammonium losses from pits (Phillips et al., 2000). EC of pig slurries was higher for the finishers, probably due to the dietary intake of salts and high protein feeding. The values in the studied area are similar to those observed by other researchers (Scotford et al., 1998a,b; Piccinini and Bortone, 1991; Kirchmann and Witter, 1992). The redox potential (Eh) values indicate

the anaerobic conditions, being due mainly to the high organic charge. The specific density (D) of slurries varied over a very low range (1004 to 1036 kg m^{-3}), this aspect being very important for accurate estimation of nutrient content. Sedimentable solids (SS) is a very simple parameter not usually used, the average value being 430 mL L^{-1} . The production stage did not affect this parameter. Total solids (TS) is one of the first easily determined parameters used to estimate the NPK content in agricultural slurries; TS values for the weaners and finishers were very similar to other countries (Zhu et al., 2003). However, for gestating and farrowing sows, the TS values were comparatively low; the high variability of slurries meant that differences were not statistically significant. The organic charge (BOD_5 and COD) of the pig slurries of the studied area were not very high, compared to the values reported by Rulkens et al. (1998) as being indicative for Dutch pig slurries (120 kg m^{-3}), probably due to management practices and the high residence time in the Spanish pits.

Nitrogen is the most difficult of the three principal nutrients to estimate by indirect correlation methods due to its variety of forms in slurries, especially considering that total nitrogen is not particularly useful in determining the amount of nitrogen available to plants (Smith et al., 1993). Chambers and Smith (1992) reported that soil mineral nitrogen measurements, following pig and cattle slurry and farmyard manure applications, were related significantly to the amount of ammonium nitrogen supplied by the manures. The average of total nitrogen for combined slurries was 2.58 kg N m^{-3} , ranging between 1.80 kg N m^{-3} , for farrowing sows, and 3.42 kg N m^{-3} , for finishers. The ammoniacal nitrogen in slurries was usually high, in

Table 1
Mean (\bar{x}) and standard deviation (\hat{s}) values of the studied pig slurries, according to the production stage, and CPS

Property	Average CPS ^A	Gestating s.	Farrowing s.	Weaners	Finishers	F-ANOVA ^B
	$\bar{x} \pm \hat{s}$	$\bar{x} \pm \hat{s}$	$\bar{x} \pm \hat{s}$	$\bar{x} \pm \hat{s}$	$\bar{x} \pm \hat{s}$	
pH	7.43 ± 0.31	7.77 ± 0.19 b	7.64 ± 0.16 b	6.88 ± 0.63 a	7.54 ± 0.34 b	***
Electrical conductivity (EC) (dS m^{-1})	17.9 ± 8.1	15.6 ± 6.1 ab	12.8 ± 6.4 a	14.2 ± 8.3 a	25.2 ± 11.0 b	*
Redox potential (Eh) (mV)	-361 ± 72	-374 ± 70	-352 ± 134	-319 ± 75	-389 ± 41	ns
Specific density (D) (kg m^{-3})	1006 ± 11	1014 ± 12	1013 ± 9	1013 ± 8	1022 ± 14	ns
Sedimentable solids (SS) (mL L^{-1})	398 ± 454	354 ± 450	340 ± 472	646 ± 556	347 ± 406	ns
Total solids (TS) (%)	2.27 ± 3.08	1.46 ± 1.73	1.69 ± 3.11	2.72 ± 3.41	3.10 ± 4.13	ns
BOD_5 ($\text{g O}_2 \text{ L}^{-1}$)	14.2 ± 9.4	11.7 ± 13.4 a	9.0 ± 5.0 a	25.0 ± 23.6 ab	21.6 ± 12.4 b	*
COD ($\text{g O}_2 \text{ L}^{-1}$)	31.6 ± 20.8	30.8 ± 36.2	24.0 ± 15.2	65.2 ± 66.1	56.4 ± 39.0	ns
Total nitrogen (TKN) (kg N m^{-3})	2.58 ± 1.29	2.35 ± 1.09	1.80 ± 0.88	2.30 ± 1.25	3.42 ± 1.75	ns
Ammoniacal N (AN) (kg N m^{-3})	2.01 ± 1.06	1.93 ± 0.82	1.38 ± 0.79	1.53 ± 0.91	2.73 ± 1.51	ns
Organic N (ON) (kg N m^{-3})	0.57 ± 0.48	0.42 ± 0.46	0.41 ± 0.40	0.78 ± 0.63	0.69 ± 0.49	ns
Potassium (K) (kg K m^{-3})	2.26 ± 1.27	1.70 ± 0.80 a	1.54 ± 0.95 a	1.75 ± 1.00 a	3.46 ± 2.01 b	*
Phosphorus (P) (kg P m^{-3})	0.76 ± 1.04	0.75 ± 0.93	0.39 ± 0.58	0.63 ± 0.61	1.07 ± 1.64	ns
Calcium (Ca) (kg Ca m^{-3})	10.8 ± 17.6	10.6 ± 15.9	6.9 ± 11.0	6.3 ± 7.7	15.6 ± 28.3	ns
Magnesium (Mg) (kg Mg m^{-3})	1.12 ± 1.17	0.90 ± 1.00	0.82 ± 0.88	0.87 ± 1.10	1.64 ± 1.53	ns

*, **, *** significantly different at $P = 0.05$, 0.01 and 0.001 , respectively. Means in the same row followed by the same letter are not different for $P = 0.05$ (Tukey test).

^A Value for the property obtained by averaging the CPS values in each of the 36 farms studied.

^B ANOVA test for comparing the production stage (Gestating s., farrowing s., weaners and finishers columns).

accordance with the literature (Kirchmann and Witter, 1992; Bonmati and Flotats, 2003), ammoniacal nitrogen as a percentage of total nitrogen being 84%, 75%, 71% and 82% for gestating sows, farrowing sows, weaners and finishers, respectively. This indicates the high degree of transformation of the studied pig slurries in pits, an important issue being the ammonia losses in these slurries due to the high pH and Mediterranean climate (Phillips et al., 2000). The experimental values of total potassium were similar to those reported by Piccinini and Bortone (1991) and Scotford et al. (1998a), but were lower those of Moller et al. (2000) and Rulkens et al. (1998). The highest values for both potassium and EC were found in finishers, which is logical since most potassium in slurries will exist in the cationic form, contributing to the total ionic strength. Phosphorus-containing compounds were particularly present in the suspended fraction, ranging between 0.39 kg P m^{-3} , for farrowing sows, and 1.07 kg P m^{-3} , for finishers, in a similar way to total nitrogen, probably due to the its organic fraction-dependence of P (Rulkens et al., 1998). Calcium and magnesium are two macronutrients not usually considered in indirect estimation procedures for nutrients in slurries, but probably play an important role in the dynamics of major nutrients, especially phosphorus, and this is an important issue in Mediterranean conditions.

3.2. Correlations to estimate plant nutrients

In Table 2, the significance levels of Pearson correlations between easily determined parameters and plant nutrients are shown for the combined pig slurries. Eh and pH seemed not to be correlated with any nutrient content. D and EC were the best easily determined parameters for estimation of nitrogen, phosphorus and potassium in the combined pig slurries. EC could be a feasible indicator for ionic species in slurries, such as

ammonium, potassium, chloride and sodium (data not shown), but is not as good for organic-dependent parameters, such as P or ON, or total contents of polyvalent cations such as Ca^{2+} or Mg^{2+} . Stevens et al. (1995) found that, in cattle and pig slurries, NH_4^+ was the dominant cation with K^+ second in importance, on a molar basis, and the concentration of each of these cations was significantly correlated with EC. Van Kessel et al. (1999) reported that specific density was the only single, easily determined parameter that can measure both total nitrogen and total phosphorus. However, density might be affected by the dilution factor, which depends on farm management. Zhu et al. (2003) also found high correlations between D and total nitrogen and phosphorus in pig slurries diluted with water to generate liquid slurries with 10 different solids contents. Additionally, in our study, an alternative derived parameter, sedimentable solids (SS), could be an inexpensive and easy determination useful for estimating total P content in slurries (Table 2).

In Table 3, selected single regression equations for nutrients are presented. In Table 4, best-fit multiple regression coefficients for nutrients in combined pig slurries are shown.

3.2.1. Nitrogen

TKN was correlated highly with EC (Fig. 1), D, BOD_5 and COD ($P < 0.001$). AN was mainly correlated with EC, BOD_5 and COD ($P < 0.001$), and D ($P < 0.01$). ON was correlated mainly with D, BOD_5 and COD ($P < 0.001$), SS ($P < 0.01$) and EC and TS ($P < 0.05$). Smith et al. (1993) reported that AN provides the best approximation to plant-available nitrogen. In our experiment, EC is the best single easily determined property for regression with AN, with a correlation coefficient, r , of 0.9144 (Fig. 2); correlation with D was not as good, mainly due to the cationic nature of ammonium in the slurries. Taking into account the high contribution of

Table 2
Correlation matrix between the different properties of CPS with nutrients ($n = 36$)

	pH	EC	RP	D	SS	TS	BOD_5	COD	TKN-N	$\text{NH}_4\text{-N}$	Org-N	K	P	Ca
EC	ns	—	—	—	—	—	—	—	—	—	—	—	—	—
RP	**	ns	—	—	—	—	—	—	—	—	—	—	—	—
D	ns	***	ns	—	—	—	—	—	—	—	—	—	—	—
SS	*	ns	ns	**	—	—	—	—	—	—	—	—	—	—
TS	ns	ns	ns	**	***	—	—	—	—	—	—	—	—	—
BOD_5	*	***	ns	***	**	**	—	—	—	—	—	—	—	—
COD	ns	***	ns	***	**	**	***	—	—	—	—	—	—	—
TKN-N	ns	***	ns	***	ns	ns	***	***	—	—	—	—	—	—
$\text{NH}_4\text{-N}$	ns	***	ns	**	ns	ns	***	***	***	—	—	—	—	—
Org-N	ns	*	ns	***	**	*	***	***	***	ns	—	—	—	—
K	ns	***	ns	***	ns	ns	***	***	***	***	**	—	—	—
P	ns	ns	ns	***	***	***	***	***	*	ns	*	—	—	—
Ca	ns	ns	ns	***	***	***	***	**	ns	ns	ns	ns	***	—
Mg	ns	ns	ns	***	***	***	***	***	*	ns	ns	ns	***	***

*, **, ***: Significant at $P < 0.05$, 0.01, 0.001, respectively. ns: Not significant. For abbreviations, see Table 1.

Table 3
Selected single regression equations for nutrients (bilateral significance <0.001) in CPS

Nutrient (kgm ⁻³)	Equation	<i>r</i>
TKN	0.132 EC (dSm ⁻¹) + 0.243	0.886
	0.0811 D (kgm ⁻³) – 79.9	0.674
AN	0.105 EC (dSm ⁻¹) + 0.097	0.914
	0.0459 D (kgm ⁻³) – 44.772	0.494
P	0.0681 D (kgm ⁻³) – 68.431	0.804
	0.001 SS (mLL ⁻¹) + 0.2017	0.571
	0.1606 TS (%) + 0.3111	0.547
K	0.1356 EC (dSm ⁻¹) – 0.2145	0.907
	0.0781 D (kgm ⁻³) – 77.351	0.650

For abbreviations, see Table 1.

Table 4
Best-fit multiple property regression coefficients for TKN, AN, P and K in CPS

Nutrient	Regression coefficients			Parameters		Percentage of explained variance, PEV (significance)
	<i>w</i>	<i>x</i>	<i>z</i>	<i>C_i</i>	<i>C_j</i>	
TKN	0.111	0.032	–31.44	EC	D	83.3 (<i>P</i> <0.001)
AN	0.107	–0.002	2.096	EC	D	83.7 (<i>P</i> <0.001)
K	0.118	0.025	–25.509	EC	D	85.4 (<i>P</i> <0.001)
P	0.061	0.049	–60.886	D	TS	66.5 (<i>P</i> <0.001)

For abbreviations, see Table 1.

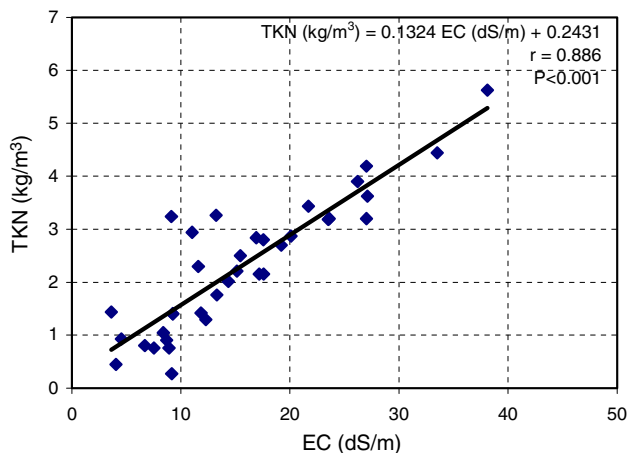


Fig. 1. Relationship between total nitrogen (TKN) and EC for combined pig slurries.

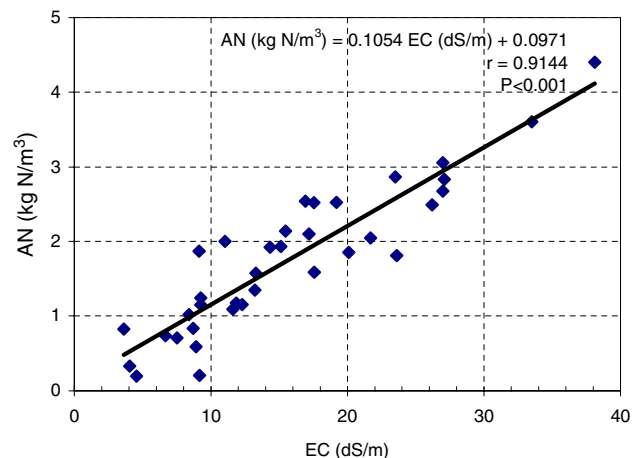


Fig. 2. Relationship between ammoniacal nitrogen (AN) and EC for combined pig slurries.

the ammoniacal form to total nitrogen (higher than 75%), TKN was correlated also with EC. However, ON was related mainly with partly dissolved or suspended parameters, like D or SS, in agreement with Rulkens et al. (1998). In the studied area, TS was not a good parameter for simple estimation of nitrogen in any form. In the multiple property regression analysis (Table 4), TKN and AN were best fitted by EC and D, with the percentage of explained variance (PEV) values higher than 83%, similar to those reported by Scotford et al. (1998).

3.2.2. Potassium

K was highly correlated with EC (Fig. 3), D, BOD₅ and COD (*P*<0.001). In our experiment, EC is the best single, easily determined property regarding regression for K (*r*=0.907), in accordance with Stevens et al. (1995); K was best-fitted by EC and D in the multiple regression analysis, with a PEV value of 85.4%, but the increment of fitting was not significant compared to the single-property EC regression. Direct determination of ammoniacal nitrogen, based in the gasometrical

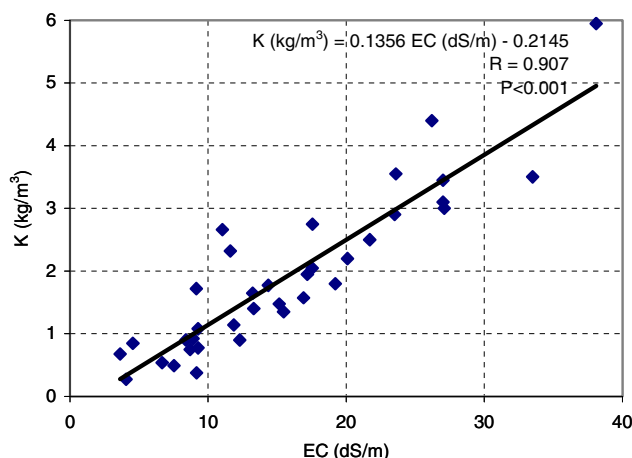


Fig. 3. Relationship between potassium and EC for combined pig slurries.

determination of molecular nitrogen (Sullivan et al., 1997) also could be a good indirect parameter for estimating potassium in slurries (Fig. 4). In our study, the regression equation was $K \text{ (kg m}^{-3}\text{)} = 1.0955 \text{ AN (kg m}^{-3}\text{)} - 0.0068$ ($r = 0.845$).

3.2.3. Phosphorus

The P content in slurries was related better to solids related parameters, such as D, SS or TS, than to EC (Table 2), as found also by Stevens et al. (1995), Scotford et al. (1998a,b), and Piccinini and Bortone (1991). However, whereas Tunney (1978) and Scotford et al. (1998b) reported a better correlation for TS, in our study D was the best single parameter for fitting total P ($r = 0.804$). In the multiple property regression analysis, a better estimation was not found using both D and TS (PEV = 66.5), probably due to the relationship between these two parameters. P seemed the worst key nutrient

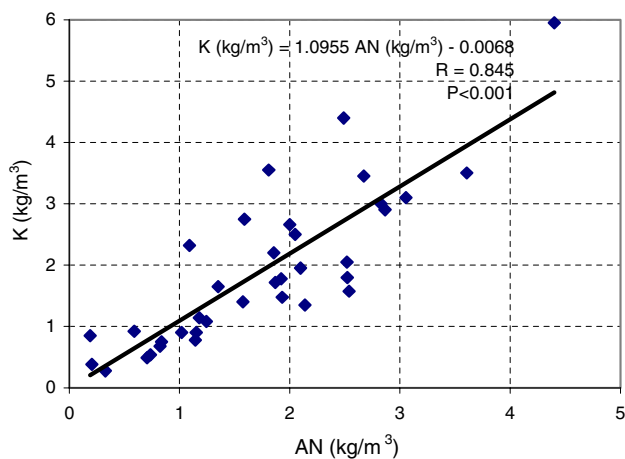


Fig. 4. Relationship between AN and potassium for combined pig slurries.

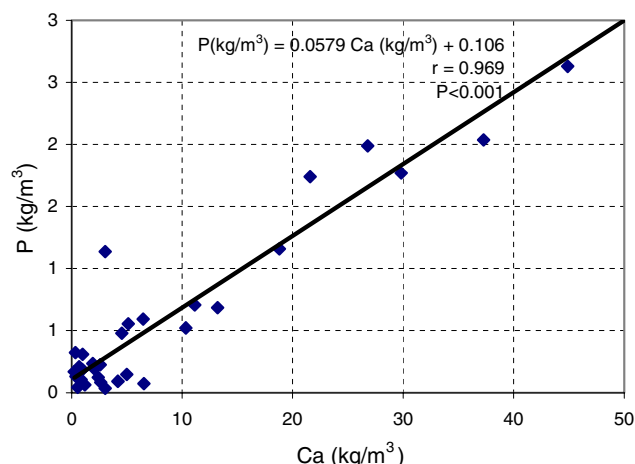


Fig. 5. Relationship between calcium and total phosphorus for combined pig slurries.

for estimation using any easily determined parameter. Similarly, for P, Zhu et al. (2003) found an estimation error higher than 50% using specific density, and PEV values ranged between 55% and 75% in other studies (Scotford et al., 1998a,b; Piccinini and Bortone, 1991).

Additionally, in our experiment, P showed a high correlation with calcium and magnesium in slurries (Fig. 5), probably due to the formation of insoluble compounds. This is an important point in slurry management for agricultural purposes in Mediterranean countries with calcareous soils and water with high contents of Ca and Mg. Thus, in calcareous soils, about 40% of total P from pig manure was fixed in non-available forms, such as insoluble calcium phosphates, a value which could reach 71% at high application rates (Bernal et al., 1993).

4. Conclusions

Characteristics of pig slurries in the studied farms in Southeast Spain were similar to those reported previously in other countries (Italy, United Kingdom, Germany, Ireland). Strong single-property relationships between electrical conductivity and total or ammoniacal nitrogen ($\text{AN (kg m}^{-3}\text{)} = 0.105 \text{ EC (dS cm}^{-1}\text{)} + 0.097$; $r = 0.914$), and potassium ($\text{K (kg m}^{-3}\text{)} = 0.1356 \text{ EC (dS cm}^{-1}\text{)} + 0.2145$; $r = 0.907$), were found. The P content in slurries was related better to solids-derived parameters such as D. In some cases (especially for ammoniacal nitrogen and potassium), the degree of improvement achieved by measuring two properties instead of one did not justify the extra expense and complication of measuring an additional property. However, the opposite was true for total nitrogen and, to a lesser degree, total phosphorus, EC-D and D-TS, respectively, being the measured slurry properties which provided best-fit relationships for those nutrients.

Therefore, electrical conductivity seemed to be the most appropriate single, easily determined parameter for estimation of total and ammonium nitrogen and potassium in pig slurries, with more than 83% of the variance explained. P seemed the worst key nutrient for estimation using any easily determined parameter. This study could contribute to a more accurate management of fertilisation with pig slurries in the studied area, in order to optimise benefits and reduce overloading with nutrients and hazardous components.

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