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https://doi.org/10.5109/1801784

出版情報：九州大学大学院農学研究院紀要. 62 (1), pp.205-212, 2017-02-24. Faculty of Agriculture, Kyushu University

J. Fac. Agr., Kyushu Univ., **62** (1), 205–212 (2017)

Development of a Quality Certification and Maturity Classification Method for Liquid Fertilizer by Measuring the Electrical Conductivity (EC) of Swine Manure

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(*Received October 20, 2016 and accepted November 4, 2016*)

The increasing need for liquid fertilizers along with the high production and treatment rate of swine manure pose a great concern in regard to the quality control and certification of fertilizers rapidly produced. In this study, 66 liquid manure samples were collected from national livestock manure co–recycling centers. These 66 samples were tested and classified into three categories based on Liquid Fertilizer Germination Index (LFGI). In addition, the physiochemical parameters of manure specimens were tested, including pH, NaCl, total sulfur (TS), total nitrogen (TN), ammonium–nitrogen (NH_4–N), nitrate–nitrogen (NO_3–N), total phosphorus (TP), potassium (K), and electrical conductivity (EC). EC had a positive correlation with TN (0.879), NH_4–N (0.816), K (0.693), and NaCl (0.625) with a significance of $p < 0.01$ but a negative correlation with LFGI (–0.719). This study demonstrated that EC could be used as an indicator in rapid testing methods for quality control to certify the maturity level of liquid manure fertilizers.

Key words: EC, liquid fertilizers, manure maturity, nutrients, quality control, swine manure

INTRODUCTION

Manure is an organic fertilizer and a valuable source of nutrients for crops to improve soil efficiency for crop production (Lorimor, 2004). If produced swain manure contain 4% of solid materials, may define as liquid manure (Lorimor, 2004).The properties and quality of manure fertilizers depend on various factors that may affect the nutrient quality of the final products. Swine manure contains huge amounts of both nutrients (macro and micro) and pollutants (heavy metals and pathogens) (Tam and Vrijmoed, 1990, 1993; Castillón, 1993; Araji *et al.*, 2001; Baloda *et al.*, 2001). These components are mostly found in an inorganic soluble form that is easy to uptake, but the amount of available nutrients are less than in composted fertilizers; thus, liquid manure fertilizers need to be applied in a huge volume for effective results (Kim *et al.*, 2013). However, uncontrolled application of these soluble inorganic nutrients as well as improperly treated manure application may cause serious soil and groundwater pollution and crop loss because of the presence of phytotoxic compounds (Alburquerque *et al.*, 2006; Butler *et al.*, 2001; Moore *et al.*, 2009; Chen *et al.*, 2007). This frequently happens when ions from the nutrient, such as nitrate and phosphorus, leach into rainwater and become stagnant underground (Márquez–Molina *et al.*, 2014).

According to a report by Kim (2014), 5.1 L/head of swine manure is produced every day, and 11,04 m^3/day of manure is recycled in public manure treatment centers in the Republic of Korea. Since manures that are used in fields have high levels of organic matter and growth of agricultural practices requires increasing amounts of fertilizers, unknown fertilizer quality may cause environmental problems (Zaha *et al.*, 2013; European Environmental Agency, 2000). Certain key manure characteristics can be used to certify the quality of the manure to minimize nutrient loss (Suresh *et al.*, 2009), improve effectiveness (Tunney and Molly 1975), and increase the rate of application; however, excess use may cause water and soil pollution (Sures *et al.*, 2009; Provolo and Martinez–Suller, 2005). Therefore, maximal use of manure fertilizers should be optimized in an economically and environmentally friendly way.

Nowadays, most manure analyses are performed following various conventional chemical testing procedures. These procedures are time–consuming and costly. Occasionally, multiple chemical tests may conflict with each other; this leads to difficulty in determining the quality of fertilizers. On the other hand, certain rapid testing techniques are widely available; however, these techniques also face some challenges relating the presentation of samples and manure sampling (Xing, 2007). Nevertheless, some notable studies have been performed using manure sampling procedures based on total nitrogen (TN) and total phosphorus (TP) (Zhu *et al.*, 2004; 2003; Ndegwa and Zhu, 2003). The relationship between electrical conductivity (EC) and nutrient contents (TN, TP, and K) can be easily determined; thus, EC could be used to estimate fertilizer quality, as indicated by Martı´nez–Suller *et al.* (2008). In a study by Stevens *et al.* (1995), there is a distinct correlation between EC and ammonium–nitrogen (NH_4–N) and K in pig and cattle slurry. Another study by Moral *et al.* (2005) showed that P is more closely correlated with solid–related parameters but has a weak and undefined correlation with EC; this was supported by Martı´nez–Suller *et al.* (2008) and Suresh *et al.* (2014). Nevertheless, the above–mentioned authors demon-

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strated in their study that EC could easily determine fertilizer value as well as quality.

EC is a measurement of the dissolved material in an aqueous solution, which relates to the ability of the material to conduct electrical current through it. The higher the dissolved material in a water or soil sample, the higher the EC will be in that material. In manure, EC reflects the degree salinity in relation to the easily decomposable compositions of manure during treatment period by assessing the total soluble ions that may or may not affect its quality as a fertilizer (Fernández *et al.*, 2007; Cardenas and Wang, 1980; Moore *et al.*, 2009; Saviozzi *et al.*, 1987). On the other hand, GI (germination index) is used to evaluate manure phytotoxicity and stability. GI is very sensitive to the nutrient contents and toxic elements of a fertilizer and defines the maturity of the manure (Oleszczuk, 2008; Zaha *et al.*, 2013). In our previous study on LFGI or Liquid Fertilizer Germination Index (Joshua *et al.*, 2016), we demonstrated a unique germination index method only for liquid manure fertilizers. During the study, we also found that EC is closely related to GI and other physiochemical properties of the specimens evaluated, and EC may be used as a quality certification tool to assess the solid compost and slurry type fertilizers in the above–mentioned studies. In addition, EC testing is a rapid and convenient test these days; thus, it could be applied onsite in a quality check technology to define the maturity of liquid manure fertilizers.

The main goal of this study was to establish EC as a regulatory indicator of quality in rapid test technologies for liquid manure fertilizers by evaluating its relationship with LFGI and other fertilizer properties in 66 liquid manure fertilizer specimens.

MATERIALS AND METHODS

Materials and storage

A total of 66 manure samples were collected from national livestock manure co–recycling centers and manure distribution centers across South Korea. The amount of manure collected was 500 mL for each specimen in a sterile plastic container. During the study period, the specimens were stored at 4°C until completion of all experiments.

Physiochemical analysis

Physiochemical analyses of pH, EC, NaCl, TN, NH_4–N, nitrate–nitrogen (NO_3–N), organic nitrogen (Org–N), TP, K, and total sulfur (TS) in the samples were performed.

Table 1. Classification of 66 specimens based on Liquid Fertilizer Germination Index (LFGI)

Sample No.	LFGI	Sample No.	LFGI	Sample No.	LFGI	Sample No.	LFGI
1	96.03	23	0.00	45	0.00	48	0.00
2	89.65	24	0.1209	46	54.85	49	0.00
3	0.00	25	0.00	47	0.00	50	0.00
4	**121.19**	26	0.1017	**mean**	**25**	51	0.00
5	109.31	27	102.67			52	0.65
6	85.50	28	118.05			53	0.09
7	90.41	29	0.00			54	0.00
8	90.12	30	0.00			55	**102.52**
9	**77.87**	31	0.03			56	0.10
10	91.52	32	24.61			57	0.01
11	79.86	33	0.00			58	**0.00**
12	102.32	34	**9.38**			59	**0.00**
13	80.67	35	28.75			60	0.00
14	87.92	36	9.51			61	0.15
15	98.29	37	64.25			62	0.09
16	100.29	38	14.90			63	0.00
17	85.25	39	0.50			64	0.03
18	108.12	40	0.14			65	0.02
19	117.44	41	0.00			66	0.06
20	79.30	42	0.01			**mean**	**5**
21	91.42	43	93.42				
22	96.31	44	92.33				
mean	**90**						

No. 1–22 (22) = matured
No. 23–47 (25) = semi–matured
No. 48–66 (19) = immature

Maturity was determined by GI. In addition, pH and EC were measured using an YSI–556MPS (xylem Inc. USA) handheld meter. NaCl was measured by the silver nitrate titration method and by the ion–electron method after LFGI treatment. TP was determined by the ascorbic acid method (APHA, 2005). TN, NH4–N, and NO3–N were analyzed by the Kjeldahl method followed by standard sewage analysis methods (JSWA, 1984). GI was determined by the LFGI method.

Statistical analysis

For data analysis, basic statistical analyses were performed to determine the maximum, minimum, mean, and standard deviation values of the fertilizer elements. SPSS software was used to determine the relationship of EC with these fertilizer elements, and we used Microsoft Office Excel 2013 for graphical purposes. However, Pearson's correlation coefficient was also used to determine the relevant relationship between EC and other physiochemical parameters as well as to demonstrate the similarity between EC and LFGI.

Table 2. Individual electrical conductivity (EC) values of 66 specimens at different maturity stages

No.	EC (mS/cm)	No.	EC (mS/cm)	No.	EC (mS/cm)
1	11.41	1	27.18	1	19.34
2	8.90	2	12.36	2	38.60
3	23.86	3	22.74	3	21.94
4	**7.03**	4	14.89	4	22.12
5	8.45	5	5.52	5	22.31
6	9.49	6	11.06	6	17.08
7	5.28	7	19.66	7	20.02
8	9.25	8	29.06	8	11.86
9	**6.41**	9	23.91	9	39.70
10	8.77	10	14.22	10	28.10
11	14.77	11	29.84	11	29.46
12	5.88	12	**18.67**	12	17.56
13	4.10	13	29.07	13	32.60
14	7.11	14	18.93	14	24.71
15	6.16	15	12.70	15	11.74
16	7.31	16	16.94	16	19.86
17	6.89	17	25.50	17	18.83
18	5.04	18	26.63	18	29.85
19	8.75	19	31.00	19	12.97
20	20.99	20	23.68	**mean**	**23.09**
21	20.91	21	13.01		
22	13.71	22	17.81		
mean	**10.02**	23	20.18		
		24	9.24		
		25	21.06		
		mean	19.79		

No. 1–22 (22) = matured
No. 23–47 (25) = semi–matured
No. 48–66 (19) = immature

RESULTS AND DISCUSSION

Determination of the maturity level of liquid manure specimens by LFGI

As described in our study on LFGI (Joshua *et al.*, 2016), we determined the maturity of 66 samples using LFGI. The samples were classified as matured (22 samples) with an average LFGI of 90, semi–matured (25 samples) with an average LFGI of 25, and immature (19 samples) with an average LFGI of 9, as shown in Table 1 and Fig. 2(A).

Relationship between EC and LFGI

Table 1 and Table 2 show the LFGI and EC of samples at different stages. Specimens with a high LFGI had a low EC value, and those with a high EC value had a low LFGI. Seed germination is highly correlated with salt stress (Joshua, 2016), and the high phytotoxicity level of manure is caused by the slow breakdown of mineral salts (Zaha, 2013). Therefore, EC and LFGI had a negative correlation as shown in Fig. 1, Fig. 2, and Table 4. Fig. 1 shows that when EC was decreased, LFGI was increased. The lowest EC value (4.1 mS/cm) was associated with a germination count of 89, and an EC value of 6.9 mS/cm was associated with the highest germination count of 121. As shown in Table 4, EC demonstrated a strong negative correlation with GI (LFGI).

EC is a measurement of the level of soluble salts from decomposing manure compounds that are critical for plant growth. EC can indicate the phytotoxicity of the compost through its salinity (Lin, 2008). Studies by Fernández *et al.* (2007), Tiquia (2005), and Saviozzi *et al.* (1987) demonstrated a good GI range at EC values of 2.0–3.0 mS/cm, where a GI correlation coefficient of –0.719 was significant at $p < 0.01$.

Chemical analysis of EC and other physiochemical properties of the manure

Differences and similarities between EC, LFGI, and potential nutrient contents of the samples were observed (Fig. 2). EC demonstrated a non–specific similarity with pH, a positive correlation with TN and NH_4–N, a partial positive correlation with K, a negative correlation with LFGI and TP, and a partial negative correlation with NO_3–N.

pH, EC, NaCl

Fig. 2 shows the level of potential nutrient contents of the samples at different maturity stages. For a better outcome, the pH range should be maintained at 6–9 (Silverstein, 2005; Eklind *et al.*, 2000; Michel *et al.*, 1998). In our study, the average and standard deviation of pH values were 7.9 ± 1.1, 8.3 ± 0.5, and 8.3 ± 1.5, with a maximum and minimum value of 8.7 and 4.6 (matured), 9.6 and 6.3 (semi–matured), and 8.9 and 7.6 (immature), respectively (Table 3). The pH conditions observed in these specimens are supported by studies performed by Suresh (2009) and Whalen *et al.* (2000), which demonstrated that the moderately high pH of manure can decrease soil acidity for manure application.

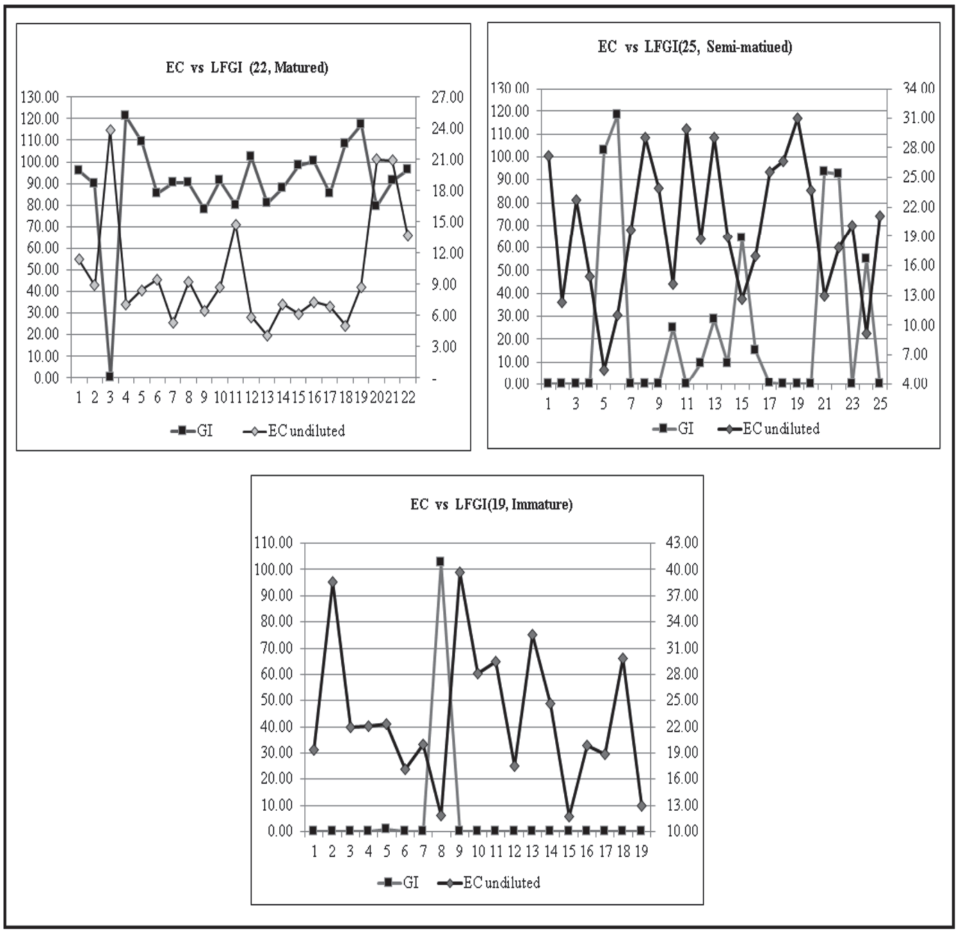

Fig. 1. Relationship (negative correlation) between EC and LFGI at different maturity stages (matured = 25; semi–matured = 22; immature = 19).

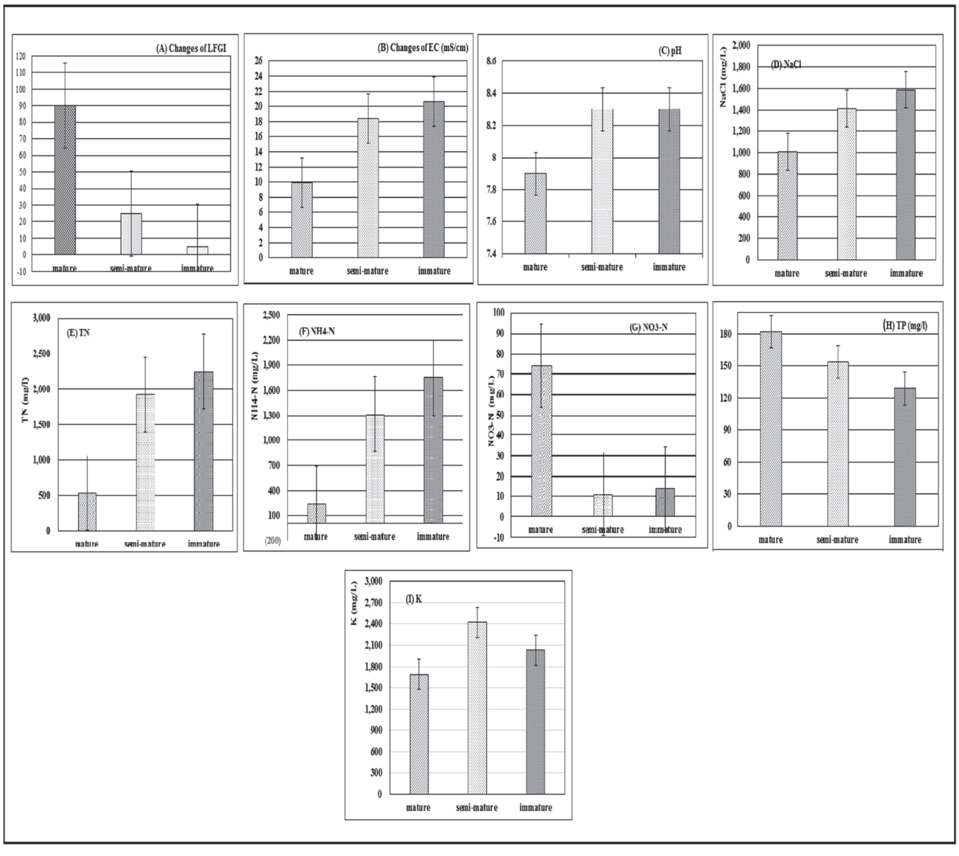

Fig. 2. Differences and similarities between LFGI, EC, pH, NaCl, NH_4–N, NO_3–N, TN, TP, and K at different maturity stages.

On the other hand, average EC values were found to be 9.9 ± 5.4 mS/cm, 18.4 ± 6.6 mS/cm, and 20.6 ± 7.3 mS/cm, with a maximum and minimum value of 23.5 and 4.1 mS/cm (matured), 29.1 and 5.4 mS/cm (semi–matured), and 35.9 and 10.3 mS/cm (immature), respectively (Table 3). These values are comparable to those in studies by Martı´nez–Suller *et al.* (2008), Moral *et al.* (2005), Scotford *et al.* (1998), and Kirchmann and Witter (1992). Although EC and pH showed a parallel growth pattern in Fig. 2, they did not have any significant relationship with each other (Table 4); this finding is similar to results of other studies (Moral *et al.*, 2005; Scotford *et al.*, 1998; Piccinini and Bortone, 1991; Kirchmann and Witter, 1992). However, NaCl showed a similar positive growth pattern with EC, having an average concentration of 1,007 ± 532.1 mg/L (matured), 1,411 ± 592.7 mg/L (semi–matured), and 1,587 ± 429.3 mg/L (immature) (Table 3 and Fig. 2).

TN, NH_4–N, NO_3–N

Among the major nutrients of plants, nitrogen is the most difficult to estimate in manure because of its variable forms (Martı´nez–Suller *et al.*, 2008; Moral *et al.*, 2005). Therefore, measurements of NH_4–N and NO_3–N are necessary. In Table 3, the properties of TN, NH_4–N, and NO_3–N are shown. TN values were 534 ± 613.8, 1,924 ± 949.6, and 2,242 ± 944.3 for matured, semi–matured, and immature stages, respectively. The observed trend could be explained by the loss of moisture during oxidation of organic matter (Huang *et al.*, 2004) in the semi–matured and immature specimens. NH_4–N values were 74 ± 93.2 (matured), 11 ± 7.9 (semi–matured), and 14 ± 8.5 (immature), and NO_3–N values were 74 ± 93.2 (matured), 11 ± 7.9 (semi–

Table 3. Physiochemical properties of 66 specimens at different maturity stages

Properties			Matured (22)	Semi–matured (25)	Immature (19)
pH	1)	Mean ± SD*	7.9 ± 1.1	8.3 ± 0.5	8.3 ± 0.5
		Max.	8.7	9.0	8.9
		Min.	4.6	6.3	7.6
	2)	Mean ± SD*	8.3 ± 1.3	8.7 ± 0.4	8.7 ± 0.1
		Max.	9.1	9.1	8.9
		Min.	4.5	6.7	8.4
EC (mS/cm)	1)	Mean ± SD*	9.9 ± 5.4	18.4 ± 6.6	20.6 ± 7.3
		Max.	23.5	29.1	35.9
		Min.	4.1	5.4	10.3
	2)	Mean ± SD*	5.1 ± 2.3	8.6 ± 2.7	9.3 ± 2.4
		Max.	10.6	13.4	14.4
		Min.	2.5	3.0	5.0
NaCl (mg/L)		Mean ± SD*	1,007 ± 532.1	1,411 ± 592.7	1,587 ± 429.3
		Max.	2,523	2,513	2,533
		Min.	387	168	931
TN (mg/L)		Mean ± SD*	534 ± 613.8	1,924 ± 949.6	2,242 ± 944.3
		Max.	2,942	3,397	4,277
		Min.	39	280	562
NH4–N (mg/L)		Mean ± SD*	238 ± 481.9	1,313 ± 766.5	1,752 ± 784.3
		Max.	2,228	2,858	3,488
		Min.	0	165	392
NO3–N (mg/L)		Mean ± SD*	74 ± 93.2	11 ± 7.9	14 ± 8.5
		Max.	426	27	36
		Min.	0	0	0
TP (mg/L)		Mean ± SD*	182 ± 163.6	154 ± 126.4	129 ± 107.7
		Max.	608	634	463
		Min.	19	18	34
K (mg/L)		Mean ± SD*	1,691 ± 857.2	2,425 ± 746.6	2,034 ± 779.9
		Max.	3,472	3,898	3,574
		Min.	88	796	34

1) Undiluted liquid manure specimens
2) After application of the LFGI pre–treatment method on the extract (filtrate)
SD* = Standard Deviation

matured), and 14 ± 8.5 (immature). According to Hirai (1983), decreasing NH_4–N level during treatment indicates high quality decomposition; this is illustrated in Fig. 2. In addition, NH_4–N and EC showed a parallel growth pattern, whereas NO_3–N and EC showed a reverse growth pattern.

TP and K

Phosphorus is one of the main components of the NPK system. This macronutrient plays an important role in regulating plant growth. Table 3 and Fig. 2 show the levels of TP in this study. Average concentrations of TP were 182 ± 163.6 mg/L (matured), 154 ± 126.4 mg/L (semi–matured), and 129 ± 107.7 mg/L (immature), and growth of TP was counter to the growth of EC. In this study (Table 3), average K concentration values were 1,691 ± 857.2 mg/L (matured), 2,425 ± 857.2 mg/L (semi–matured), and 2,034 ± 857.2 mg/L (immature) (Fig. 2), which were not sequential. That may explain why K had a negative correlation with LFGI; however, changes in K cation during decomposition demonstrated it had a positive correlation with EC.

Correlation between EC and physiochemical parameters

The significance levels of the Pearson's correlation coefficients between EC, physiochemical parameters and plant nutrient contents are shown in Table 4. It should be noted that pH was previously not correlated with any of the nutrient contents in other studies (Martı´nez–Suller *et al.*, 2008; Moral *et al.*, 2005). EC could be an effective measurement indicator of ionic species in manures, such as ammonium, nitrate and potassium (Martı´nez–Suller *et al.*, 2008; Moral *et al.*, 2005). NH_4^+ is a dominant cation that breaks down to NO_3^-; as a result, EC can have a significant positive correlation with NH_4–N (Stevens, 1995) and negative correlation with NO_3–N. For the same reason, it can also have a similar correlation pattern with K^+; however, EC is not a good indicator of organic parameters such as Org–N and Org–P (Márquez–Molina *et al.*, 2014).

TN, NH_4–N, Org–N, NO_3–N

There was a significant high positive correlation ($p < 0.01$) between EC and TN, NH_4–N, and Org–N at 0.879, 0.816, and 0.720, respectively. On the other hand, NO_3–N was significant at $p < 0.005$, demonstrating a weak negative correlation with EC at –0.315. In Fig. 3(B, C, D, E), the regression equation corresponding to three nitrogen components are shown with their correlation with EC. Here, regression analyses of pig manures were highly significant at $p < 0.01$ for the nitrogen compounds. TN had the highest determination coefficient with EC at $r^2 = 0.7718$ [regression equation, TN (mg/L) = 124.53 (EC (mS/cm)) – 468.05]. The determination coefficient of NH_4–N was $r^2 = 0.6653$ [regression equation, NH_4–N (mg/L) = 95837 (EC (mS/cm)) – 473.47] and Org–N was $r^2 = 0.5182$ [regression equation, Org–N (mg/L) = 29.459 (EC (mS/cm)) –56.61]. On the other hand, the determination coefficient of NO_3–N was $r^2 = 0.1594$ [regression equation NO_3–N (mg/L) = –44.941ln (EC (mS/cm)) + 152.21]. These findings are similar to the results of studies by Suresh (2009), Martı´nez–Suller *et al.* (2008), and Moral *et al.* (2005).

TP

EC did not have significant correlation with TP (0.289, $p < 0.005$) (Table 4). The regression equation was TP (mg/L) = 4.939 EC (mS/cm) + 76.137, and the determination coefficient was $r^2 = 0.5182$ (Fig. 3). P is associated with manure struvite as a solid fraction. Studies have suggested that P contents are better indicated by solid–related parameters (Moral *et al.*, 2005; Scotford *et al.*, 1998; Stevens *et al.*, 1995; Piccinini and Bortone, 1991). Therefore, estimating P based on EC may not be accurate (Provolo and Martinez–Suller, 2007).

K

The correlation between K and EC was high (0.693, $p < 0.01$) (Table 4). K was easily defined by EC; the determination coefficient was $r^2 = 0.4798$, and the regression equation was K (mg/L) = 74.108 EC (mS/cm)

Table 4. Correlation coefficients between major physiochemical properties in matured liquid fertilizer

Properties	pH[1]	EC[1]	EC[2]	NaC[1]	TN	NH_4–N	NO_3–N	Org–N	P	K	GI
pH[1]	1										
EC[1]	0.139	1									
EC[2]	0.128	0.980**	1								
NaC[1]	0.077	0.625**	0.626**	1							
TN	0.367**	0.879**	0.875**	0.550**	1						
NH_4–N	0.365**	0.816**	0.823**	0.517**	0.961**	1					
NO_3–N	–0.385**	–0.315*	–0.327**	–0.355**	–0.292*	–0.302*	1				
Org–N	0.293*	0.720**	0.701**	0.402**	0.737**	0.550**	–0.310*	1			
P	–0.521**	0.289*	0.257*	0.254*	0.186	0.137	0.040	0.216	1		
K	0.090	0.693**	0.667**	0.736**	0.567**	0.459**	–0.337**	0.613**	0.435**	1	
GI[3]	–0.393**	–0.719**	–0.724**	–0.402**	–0.826**	–0.803**	0.324**	–0.582**	–0.001	–0.385**	1

*significant at $p < 0.005$; **significant at $p < 0.01$; [1] before LFGI dilution; [2] after LFGI dilution; [3] LFGI

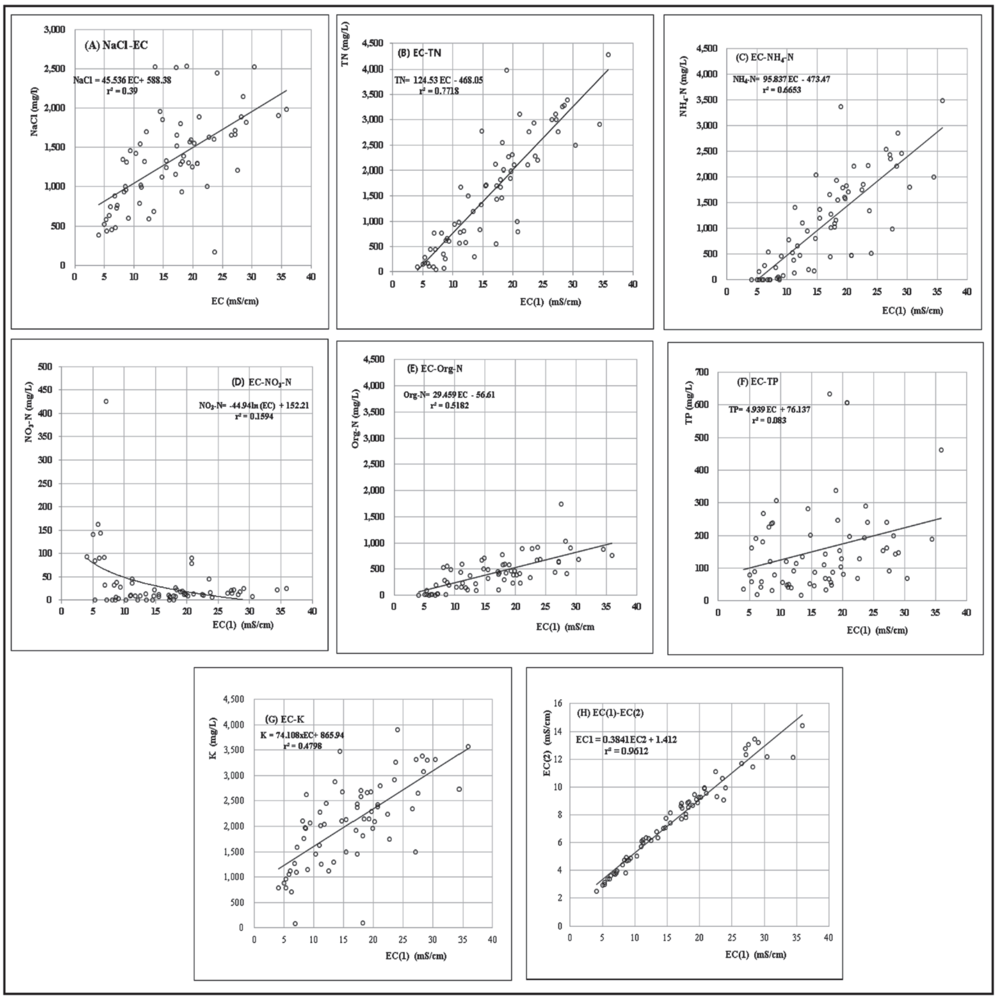

Fig. 3. Correlation between raw electrical conductivity (EC) values of manure samples and other physiochemical properties (NaCl, TN, NH_4–N, NO_3–N, K, and EC [diluted]).

+ 865.94 (Fig. 3). K has been demonstrated to be highly correlated with EC by Stevens *et al*. (1995) and Moral *et al*. (2005). This suggests the feasibility of measuring K in manure by EC.

NaCl (salt)

As mentioned previously, EC values reflect the degree of salinity in manure (Varma and Kalamdhad, 20113); this explains the strong correlation of EC with NaCl (0.625), which was significant at $p < 0.01$ (Table 4). The determination coefficient was $r^2 = 0.39$, and the regression equation of NaCl was NaCl (mg/L) = 45.536 EC (mg/L) + 588.38.

CONCLUSION

Quality measurement of liquid manure is necessary to ensure the effective use of manures and develop new technologies for environmental safety. The physiochemical properties and maturity degree (LFGI) of manure samples were assessed in this study. EC was found to be a suitable indicator of nutrient contents and GI. EC showed a strong positive specific correlation with TN, NH_4–N, Org–N, and K and a strong negative correlation with GI (LFGI). The efficiency of EC as a quality prediction tool has been demonstrated for animal compost and slurry type fertilizers. This study revealed that EC could be used as an indicator in rapid testing methods for quality control based on the correlation of EC with nutrient contents. For improved validation, both LFGI and EC may be used for the same purpose. Furthermore, the EC value for optimal correlation was determined to be between 10.3 mS/cm and 4.1 mS/cm (Table 3), which is in–between the recommended EC level (15 mS/cm) of the LFQC certification system. According to previous studies and the present study, EC could be conveniently used as a parameter in rapid testing methods for quality control of any types of liquid manure before use as a fertilizer.

ACKNOWLEDGMENTS

This research was funded by the Ministry of Agriculture, Food and Rural Affairs (MAFRA) (project # 316024–3" Livestock Manure Nutrient Management and Business Model"). In particular, we express a very special and sincere thanks to Prof. Yabe, Kyushu University, Japan for his valued collaboration in the experimental design and interpretation of the results of this study and for his help in writing this research paper.

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