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THE EFFECT OF PIG DIET AND SOLID/LIQUID SEPARATION OF PIG SLURRY ON PHOSPHORUS FRACTIONATION

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ABSTRACT

In some intensive animal production areas, the accumulation of nutrient surpluses (N, P,…) from livestock effluents has led to severe pollution problems (water, air, soil).

The control of this potential pollutant load requires the development of processing methods to remove the excess nutrients. In France, biological treatment based on aeration (nitrification/denitrification) is the most widespread technology on farms. Nowadays this treatment has to be adapted to include phosphorus removal as well as nitrogen removal.

For this purpose, the characteristics of pig slurry from different farms (with or without phytase in pig diets) were studied through phosphorus, magnesium and calcium fractionation including ortho-P, organic dissolved P, precipitated P, biomass-P and residual-P. Moreover, the influence of mechanical separation (press-auger and centrifugation) was studied.

In raw slurry, 4-10 % of phosphorus was soluble, 60-85% was precipitated and 3-20% was phosphorus linked to the biomass. The total phosphorus concentration is slightly decreased when diets with phytase are used (12%). Without phytase, around 20% of total phosphorus was "residual", i.e. on a very insoluble form, probably as calcium phytate. Up to 50% of this form remained after the separation step. No "residual" phosphorus was found with phytase in the diet.

Both separators studied (press auger and centrifugation) did not affect the concentration of soluble compounds in the separated slurry. When TSS concentration in raw slurry is high (>3,5%), the abatement of the TSS concentration is similar with centrifugation or press-auger. In contrast, centrifugation and press-auger decreased the total phosphorus concentration in the effluent by up to 50% and 15% respectively. Most of the phosphorus removed by centrifugation is precipitated phosphorus.

The difference between the amount and the quality of the phosphorus present in the effluents for each type separator should be considered in proposing a relevant dephosphorisation strategy for pig slurry.

KEYWORDS. animal wastes, phosphorus recycling, biological treatment, characterization, liquid manure, magnesium, calcium.

INTRODUCTION

In intensive animal production areas, nitrogen and phosphorus in livestock wastes are higher than crop requirements. Nitrogen is the major concern with regard to European regulation. However, in France, phosphorus is now also considered and must be removed from the slurry when nitrogen is removed to maintain the ratio N/P in the product of the treatment. In order to comply with law, several approaches have been developed. For example, the use of microbial phytase in diet improves the digestion of P-phytate which is the main phosphorus form in grain diets and allows the minimization of the mineral-P supplementation. P excretion is reduced by 20-55% depending on the growth phase (Roberson, 1999 ; Jongbloed et al., 2000 ; Grandhi, 2001).

In France, to avoid nitrate contamination in groundwater, biological treatment of pig slurry by intermittent aeration (nitrification/denitrification) has been developed with about 300 treatment units installed on pig farms. Mechanical separation by press-auger ahead of the biological reactor is more prevalent. This method allows 15-25% removal of total phosphorus in the solid phase (Burns and Moody, 2001 ; Daumer et al., 2001). Mechanical separation by centrifugation is the common technology used when more phosphorus removal is needed. It is often combined with biological treatment for both phosphorus and nitrogen removal. The solid product of this separation step contains 50-80% of the initial phosphorus of the pig slurry. Even after composting, this product is still a good organic fertilizer with a high phosphorus content, its agronomic value is compromised due to its high heavy metal content (Béline et al., 2003).

Treatment options have to be improved to remove and recycle phosphorus in a sustainable manner, apart from sludge and solids containing heavy metal. The first step in developping a new strategy for phosphorus recycling is to characterize the different forms of phosphorus in the influent to be treated.

The objectives of this work were i) to quantify the influence of phytase in pig diets on the phosphorus forms in raw slurry at a farm level, ii) to evaluate the influence of the preliminary solid/liquid separation step on distribution of the different forms of phosphorus, and iii) to discuss the ability of the different forms to enter a biological or a physical dephosphorisation process.

MATERIAL AND METHODS

Slurry :

Raw and separated slurries were sampled in 4 different farm-scale treatment plants in Brittany (NW of France). Characteristics of the farms and plants are depicted in Table 1 and Figure 1.

Table 1 : Farms and plants characteristics

	Sows	Weaning pigs	Fattening pigs	type of diets	separation equipment
		places			
F1	392	1800	1592	commercial with phytase	centrifugation (rated flow max: $6m^3.h^{-1}$)
F2	420	1226	2230	commercial with phytase	press-auger (0,5 mm grid + decantation and settling sludge recirculation, HRT = 5 days)
F3	420	1440	2960	homegrown without phytase	centrifugation (rated flow : 16 $m^3.h^{-1}$)
F4	250	1150	1850	commercial without phytase	press-auger (0,7 mm grid)

For each experiment, two samples of raw slurry (RS) were collected in the reception pit after 30 minutes of mixing, twice a month over 3 months. In the same way, two samples of separated slurry (SS) were sampled as output from the mechanical separation equipment.

The fractionation method with perchloric acid (PCA) described by Appelddorn (1992) for wastewater sludge was adapted as shown in figure 2.

Laboratory analyses :

Total solids (TS) , total suspended solids (TSS), volatile solids (VS) and volatile suspended solids (VSS) were analyzed using the standard methods (1992).Total phosphorus was measured by a flow injection analyzer (Lachat Instrument, Milwaukee, WI, USA) with a blue molybdate method after digestion with peroxodisulfate and sulfuric+nitric acid at 120°C. Dissolved ortho-phosphate in slurry and PCA extract (colored samples) were analyzed by ionic chromatography. All the samples were analyzed for magnesium and calcium by ionic chromatography (DIONEX, Sunnyvale, CA, USA). Results for each sample are the mean of two analyses.

Figure 1 : Separation devices (Figure 1 not available)

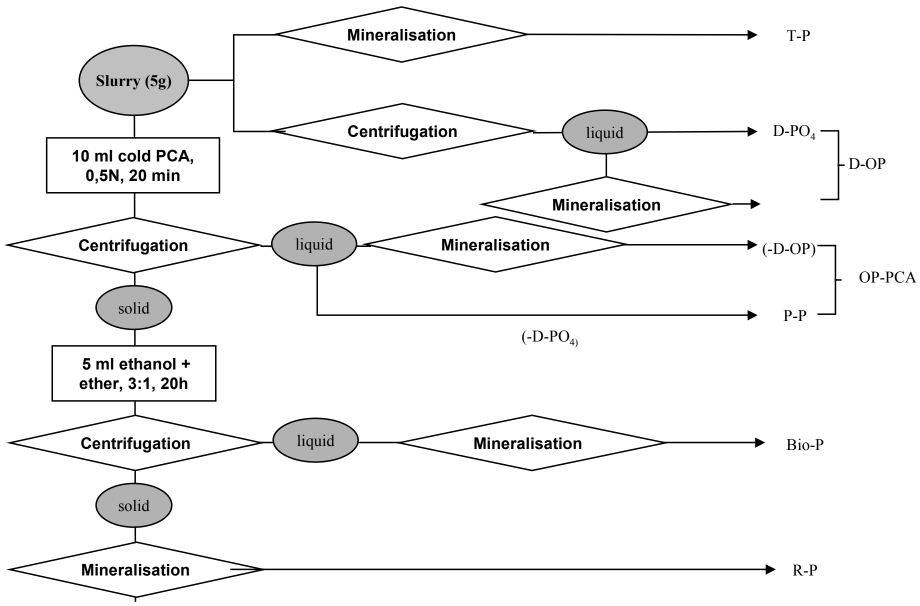

Figure 2 : Fractionation method : T-P : Total phosphorus ; $D\text{-}PO_4$: dissolved orth-phosphate ; D-OP : dissolved organic-phosphate ; OP-PCA : organic phosphate from PCA fraction ; P-P : precipitated phosphorus ; Bio-P : biomass phosphorus ; R-P : residual phosphorus

Results and Discussion

Raw slurries characteristics

DM and TSS content varied from one farm to another but also varied with sampling time (Table 2). Farm 3 had a regular and diluted slurry, while farm1 had a concentrated and variable slurry.

Table 2 : Limit values observed in raw slurry for dry matter (DM) and total suspended solids (TSS)

	F1		F2		F3		F4	
number of samples	6		6		4		4	
	min	max	min	max	min	max	min	max
DM ($mg.g^{-1}$)	42	66	33	48	25	26	43	55
TSS ($mg.g^{-1}$)	35	62	24	38	15	15	33	44

These characteristics depend on physiological stages of the pig growth, feeding and cleaning practices in the building. Even if the retention time in the homogenization tank is long (up to 18 days) it is not sufficient to obtain a regular influent in the treatment process. In the following paragraphs, all the characteristics will be expressed as a function of DM to discard the dilution effect.

Phosphorus, calcium and magnesium in raw slurries

Two samples from each farm were used for complete phosphorus analysis. The total phosphorus content is lower at farms that used phytase (12%)(Table 3). Phytase increased the digestibility of phosphorus from the diet and allowed to reduce phosphorus excretion depending on the amount and activity of the enzyme, the composition of the diet and the growing phase. Reduction from 18 to 50% have been cited in literature (Roberson, 1999 ; Dou et al., 2000 ; Jongbloed et al., 2000 ; Grandhi, 2001).

For all slurries tested, dissolved phosphorus (6-8%) was mainly as ortho-phosphate and 60-80% of phosphorus is in a precipitated form. These results are in agreement with results obtained by several authors using comparable different fractionation methods (Gerritse and Zugec, 1977 ; Leinweber, 1996 ; Dou et al., 2000 ; Sharpley and Moyer, 2000). In farms F1 and F2 (with phytase) remaining phosphorus after PCA extraction was bio-P and no residual phosphorus was observed. Concurrently, for the farms F3 and F4, most of the remaining phosphorus was in a residual form (Figure 3). Dou et al. (2000) obtained similar results showing that residual phosphorus was less important (12%) in slurry from pigs fed with a diet containing phytase.

Table 3 : Forms of phosphorus in raw slurries.

	F1	F2	F3	F4
	$mg.gDM^{-1}$			
$D\text{-}PO_4$	2	1	3	1
D-OP	1	0	0	1
P-P	18	21	20	17
OP-PCA	0	0	0	2
Bio-P	5	3	1	3
R-P	0	0	5	4
TP	26	25	29	28

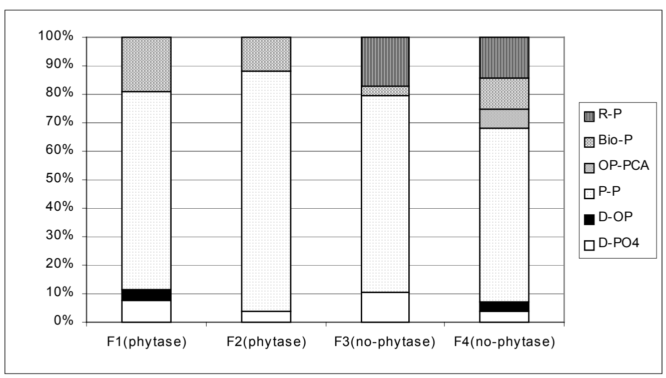

Figure 3 : Distribution of phosphorus forms in the raw slurry of four pig farms in France.

Fractionation of calcium and magnesium are shown in Tables 4 and 5. Dissolved and precipitated forms were the main form in slurry (40-70% and 78-92% respectively). Without phytase feeding, calcium was also found in the residual fraction. By contrast, even without phytase, only a small part of magnesium was in a residual form. Dissolution of calcium and magnesium by PCA showed that the soluble phosphorus in this acid phase was mainly due to dissolution of the crystallized forms and not to the mineralization of organic forms. The residual form of phosphorus could be calcium phytate which is an insoluble compound.

Table 4 : Forms of calcium in raw slurries.

	F1	F2	F3	F4
	mg.gDM^{-1}			
D-Ca	9	14	10	6
D-OCa	0	0	0	0
P-Ca	16	24	13	31
OCa-PCA	0	0	3	0
Bio-Ca	1	2	0	0
R-Ca	0	0	7	7
TCa	26	40	33	44

Table 5 : Forms of magnesium in raw slurries.

	F1	F2	F3	F4
	mg.gDM^{-1}			
D-Mg	1	1	1	0
D-OMg	0	0	0	0
P-Mg	11	12	11	14
OMg-PCA	0	0	1	4
Bio-Mg	0	1	0	0
R-Mg	0	0	1	0
TMg	12	14	14	18

Phytic acid, which is the main phosphorus form in cereal grain diets, may form complexes with calcium added to the diet and/or protein and inhibit enzymes, reducing the degradation of the diet. Through the degradation of the phytic acid, phytase could avoid the formation of calcium phytate and increase the degradation of the residual fraction by keeping the enzymatic activity intact.

<u>Efficiency of the separation step</u>

In farms, the separation step is optimized not only to increase solid and nutrient removal but also to obtain a solid product that can be composted easily (DM ≥ 300 mg.g^{-1}). The remaining TSS in separated slurry from centrifugation was between 4 and15 mg.g^{-1}. The TSS abatement by centrifugation (65-78%) was greater when the TSS content of the raw slurry was above 40 mg.g^{-1}. The least abatement (33%) was observed for diluted slurry (TSS in raw slurry = 15 mg.g^{-1}). A similar influence of the initial TSS was shown by Piccini and Cortellini (1987) and Sneath et al. (1988) for centrifugation of pig slurry without flocculent. In farm F4, which had the most basic separation device, remaining TSS concentration was higher (24-30 mg.g^{-1}) and the abatement of

TSS was always under 30% even though the TSS concentration in raw slurry was over 40 $mg.g^{-1}$. TSS abatement by the separation step from farm F2 (37-77%) was nearly the same as by centrifugation but residence time of raw slurry in the system (5 days), decantation and recirculation did not allow to relate the efficiency of TSS removal and the initial TSS content.

By contrast, phosphorus removal efficiency depended on the type of device. In this trial the TSS abatement was 65% for F1 (centrifugation), and 64% for F2 (the press-auger). However, the total phosphorus concentration was decreased by 52% in F1 and only 15% in F2. Raw slurry from farm F3 had a low TSS content (15 $mg.g^{-1}$) . The solid removal efficiency was only 33% but phosphorus concentration was decreased by 53%. For farm F4, TSS was reduced by 28% and phosphorus concentration in separated slurry increased (+20%) (Figure 4).

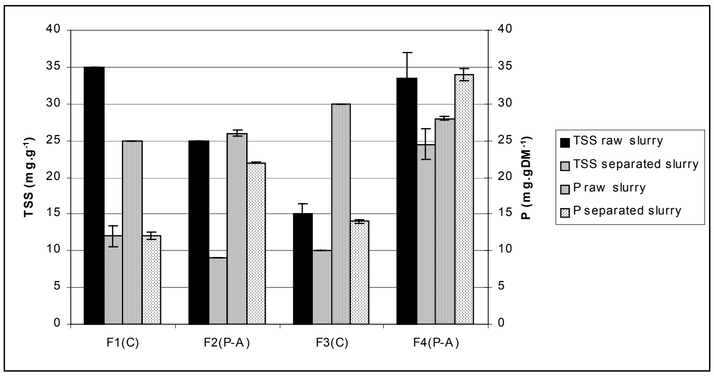

Figure 4 : TSS and total phosphorus concentration in slurry before and after the separation step. (C : centrifugation ; P-A : press-auger)

These figures show that phosphorus content of the solids removed by both systems were not the same. Centrifugation could separate small and dense particles which were not retained by the grid of the press- auger. These small particles represent a low mass fraction (Stambouli, 1974 ; Sneath et al., 1988) but have a high phosphorus content (Gerritse and Zugec, 1977). On the other hand, the press-auger from farm F4 separated only large particles which have a low phosphorus content.

The same argument could be used to explain the results observed for calcium and magnesium separation (Figure 5). Calcium, which was not retained by the press-auger of farm F4 was eliminated slightly better by centrifugation than by the press-auger of farm F2. Calcium could be mainly in the form of smaller and/or less dense particles than phosphorus.

Like phosphorus, magnesium separation efficiency, was distinctly higher with centrifugation than with the press-auger. Magnesium is likely to be trapped in the small and dense particles.

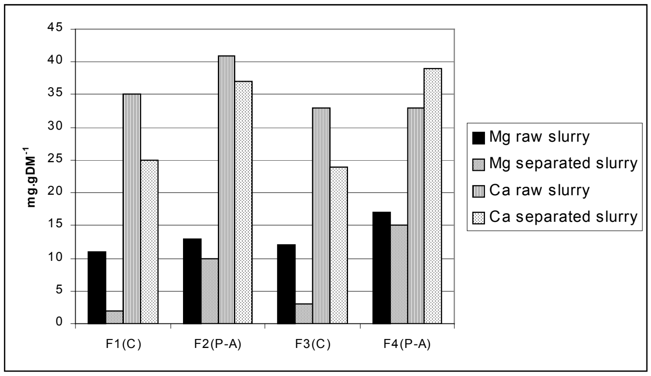

Figure 5 : Mg and Ca concentration in slurry before and after the separation step

Characterization of the effluent.

The Table 6 shows the concentration of the different forms of phosphorus in the effluents from the four farms.

Table 6 : Forms of phosphorus in separated slurries.

	F1	F2	F3	F4
	$mg.gDM^{-1}$			
$D\text{-}PO_4$	4	2	4	2
D-OP	0	1	1	1
P-P	3	14	7	30
OP-PCA	2	1	0	0
Bio-P	4	2	0	0
R-P	0	0	3	3
TP	13	21	15	35

The distribution of phosphorus was nearly the same before and after the separation step by the press-auger. Precipitated and dissolved P represents 70-85% and 5-10% of total phosphorus, respectively (Figure 6).

Centrifugation removed mainly the precipitated form which represented only 20-50% of the total phosphorus in the effluent. The residual phosphorus concentration still represented 22% of the total phosphorus in the effluent of the centrifugation step in farm F3 (Figure 6)

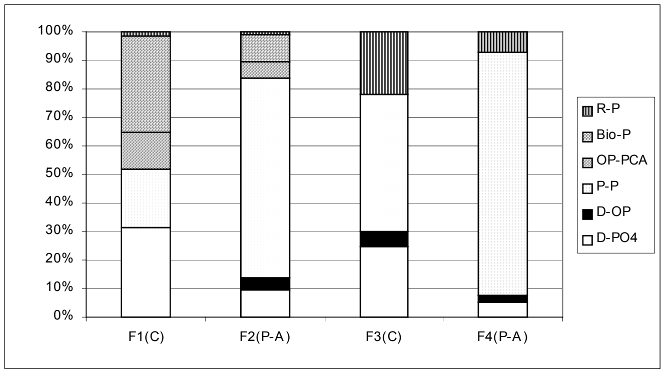

Figure 6 : Distribution of phosphorus in separated slurries in four pig farms in France.

<u>Consequences on further treatment :</u>

Effluents from centrifugation had a low phosphorus content. In this case, a simple way to recycle phosphorus could be through the optimization of the current nitrification/denitrification biological treatment in order to immobilize phosphorus in the sludge phase. This could be realized by precipitation and/or biological immobilization by the dephosphorisation processes developed for wastewater treatment. Results from many authors have shown that the enhancement of phosphorus removal from raw or separated slurry is possible by continuous or intermittent aeration. Both precipitation and biological removal took place in the same reactor (Bicudo and Svoboda, 1995 ; Maekawa and al., 1995 ; Bernet et al., 1998, tilche et al., 1999 ; Luo et al., 2001 ; Ndegwa et al., 2001 ; Zhu et al., 2001 ; Luo et al., 2002 ; Suzuki et al., 2002). However, further agronomic management of the solids obtained from the centrifugation step are still difficult due to its high heavy metal content (Béline et al., 2003).

Effluents from press-auger separation had a very high phosphorus content. In this case, phosphorus content in the sludge from the biological treatment of such an effluent is too high to use them in a sustainable agronomic way. One way to recycle phosphorus could be to partially dissolve the precipitated phosphorus and, after a decantation or a centrifugation step, to separate phosphorus from the supernatant by precipitation (without organic matter or heavy metal). The dissolution could be reached by modifying the current nitrification /denitrification process in order to either decrease the pH or to optimize the phosphorus release by a biological process. Greaves (2001) observed such a release by intermittent aeration on a diluted slurry.

CONCLUSIONS

1. Phytase in diets seemed to decrease the amount of residual phosphorus which are very insoluble forms (probably calcium phytate). However, further works are needed to confirm this hypothesis. The impact of the phytase on the phosphorus forms in slurry had never been studied before and our results are only from two different farms.
2. Centrifugation is more efficient than press-auger for phosphorus abatement probably by separating small and dense particles with a high phosphorus content. The form removed by centrifugation is mainly the precipitated form. However, management of the solid from the centrifugation step is still difficult due to its high heavy metal content.
3. The effect of the separation step on the distribution of magnesium is nearly the same as for phosphorus. Centrifugation is less effective for calcium than for phosphorus or magnesium removal.
4. Due to quantitative and qualitative differences of the effluent, different strategies should be developed to recycle phosphorus from the press-auger and centrifugation way.

REFERENCES

1. Appeldoorn K. J., Boom A. J., KOrstee J. J. and Zehnder A. J. B. 1992.Contribution of precipitated phosphates and acid-soluble polyphosphate to enhanced biological phosphate removal. *Wat.Res.*,26(7):937-943
2. Béline F., Daumer M-L., Guiziou F. 2003. Performances des principales filières de traitement biologique aérobie des lisiers de porcs. In *35 emes journées de la Recherche Porcine*. 4,5,6 february 2003. Paris, France *(in press)*
3. Bernet N., Delgenes J. P. and Moletta R.1998. A new anaerobic-aerobic process for treatment of piggery wastewaters in two coupled sequencing batch reactor. In *European conference on new advances in biological nitrogen and phosphorus removal for municipal or industrial wastewaters* 261-264.INRA.Narbonnne, France.
4. Bicudo J. R. and Svoboda I. F. 1995.Effects of intermittent-cycle extended-aeration treatment on the fate of nutrients, metals and bacterial indicators in pig slurry. *Bioresource Technology*,54(63-72):
5. Burns R. T. and Moody L. B.2001. Performance testing of srew-press solid separators : Comprehensive solids analysis and nutrient partitioning. In *International symposium adressing animal production and environmental issues*, Raleigh, N.C.
6. Daumer M.-L., Béline F. and Guiziou F.2001. Biological treatment of pig slurry in France : process performance and co-products characterization. In *ORBIT 2001, Organic Recovery and Biological treatment*, 245-252.Orbit Association. Seville.

7. Daumer M.-L.2001. Phosphate recovery following biological aerobic treatment of pig slurry. In Second international conference on Recovery of Phosphates from sewage and animal wastes. CEEP. Noordwijkerhout, Holland.

8. Dou Z., Toth J. D., Allshouse R., Ramberg C. F. and Ferguson J. D.2000. Phosphorus fraction distribution in animal manure. In *8th international symposium of ASAE*, 1-9.J. A. Moore. Des Moines, IOWA.

9. Gerritse R. G. and Zugec I. 1977. The phosphorus cycle in pig slurry measured from 32PO4 distribution rates. *J.Agric. Sci*. Camb.,88:101-109

10. Grandhi R. R. 2001.Effect of supplemental phytase and ideal dietary amino acid ratios in covered and hulless-barley-based diets on pig performance and excretion of phosphorus and nitrogen in manure. *Canadian Journal of Animal Science*,81(1):115-124

11. Greaves J., Haygarth P. and Hobbs P.2001. A novel biological phosphate pump. In *Second international conference on Recovery of Phosphates from sewage and animal wastes*. Noordwijkerhout .Holland.

12. Jongbloed A. W., Mroz Z., van der Weij-Jongbloed R. and Kemme P. A. 2000.The effects of microbial phytase, organic acids and their interaction in diets for growing pigs. *Livestock Production Science*,67(1-2):113-122

13. Leinweber P., Haumaier L. and Zech W. 1997.Sequential extractions and P-NMR spectroscopy of phosphorus forms in animal manures, whole soils and particle-size separates from a densely populated livestock area in northwest Germany. *Biology and Fertility of Soils*,25(1):89-94

14. Luo A., Zhu J. and Ndegwa P. M. 2002.Removal of carbon, nitrogen and phosphorus in pig manure by continuous and Intermittent aeration at low redox potentials. *Biosystems Engineering*,82(2):209-215

15. Maekawa T., Chung-Min L. and Xing-Dong F.1995.Nitrogen and phosphorus removal for swine wastewater using intermittent aeration batch reactor followed by ammonium crystallization process.*Wat.Res.*,29(12):2643-2650

16. Roberson K. D. 1999.Estimation of the phosphorus requirement of weanling pigs fed supplemental phytase. *Animal Feed Science and Technology*,80(2):91-100

17. Roberson K. D. 1999.Estimation of the phosphorus requirement of weanling pigs fed supplemental phytase.*Animal Feed Science and Technology*,80(2):91-100

18. Sharpley A. and Moyer B. 2000.Phosphorus forms in manure and compost and their release during simulated rainfall. *J.Environ.Qual*.,29(5):1462-1469

19. Sneath R. W., Shaw M. and Williams A. G. 1988.Centrifugation for separating piggery slurry. 1.The performance of a decanting centrifuge. *J.Agric. Engng Res.*,69:115-125

20. Stambouli N. and Ballay D.1974. Répartition de la charge polluante du lisier de porcherie en fonction de ses différents constituants physiques. In *Journées de la recherche porcine en France*, 93-100.INRA-ITP.

21. Suzuki K., Tanaka Y., Osada T. and Waki M. 2002.Removal of phosphate, magnesium and calcium from swine wastewater through crystallization enhanced by aeration. *Wat.Res.,*36(12):2991-2998

22. Tilche A., Bacilieri E., Bortone G., Malaspina F., Piccinini S. and Stante L. 1999.Biological phosphorus and nitrogen removal in a full scale sequencing batch reactor treating piggery wastewater. *Water Science and Technology*,40(1):199-206