Paper # NABEC/CSBE 12-058

Optimizing process of dry matter separation from swine manure by centrifugation using response surface methodology

Parminder Singh[1, 2], Stéphane Godbout[2], Robert Lagacé[1], Mausam Verma[2]

[1]Départment de sols et de génie agroalimentaire Faculté des sciences de l'agriculture et de l'alimentation, Université Laval, Québec, QC G1K 7P4, Canada

[2]Institut de recherche développement agroenvironnement Centre de recherche 2700, rue Einstein Québec (Québec) G1P 3W8, Canada

Abstract. In Québec there is a growing need to separate swine manure into solid and liquid fraction due to intensive livestock production at farms that leads to surplus production of liquid swine manure, which is difficult to transport to other farmlands. In the past, chemical additives have been used to increase separation efficiency of liquid swine manure which causes environment pollution. Therefore, in this study biochar is being used as an additive for separating liquid swine manure which is a by-product of pyrolysis. In this study the objective is to separate liquid swine manure with lower dry matter content by centrifugation in a laboratory centrifuge utilizing biochar as an additive to obtain a solid portion with higher dry matter content and a liquid portion with low dry matter content. Biochar was added in different percentage fractions of 0.64%, 2%, 4 %, 6% and 7.36% to the liquid swine manure during centrifugation to increase the centrifugal sedimentation rate. Optimization of the process was done by applying central composite design and response surface methodology using SAS program. A second order quadratic polynomial equation was obtained to predict the dry matter content as a function of percentage of biochar, pH and time. pH and biochar significantly affected the separation of dry matter content, pH being the most effective followed by percentage of biochar. The maximum response obtained for dry matter content from liquid swine manure was 33.84% (w/v) dry matter content at 6% (w/v) biochar, pH 13 and 90 min time of centrifugation.

A Community of

Keywords. Solid-liquid separation, centrifugation, biochar, swine manure

American Society of Agricultural and Biological Engineers

For presentation at
NABEC-CSBE/SCGAB 2012 Joint Meeting and Technical Conference
Northeast Agricultural & Biological Engineering Conference
Canadian Society for Bioengineering
Lakehead University, Orillia, Ontario
July 15-18, 2012

Introduction

In Quebec, there is growing interest from swine producers to remove water from manure via economically feasible methods and value addition to the separated solids. Average manure production by a swine is 7.6 litres per day (Drolet and Gilbert, 2000), which leads to production of surplus amount of swine manure at the farms with low dry matter content due to which retention of manure at farms is problematic.

Transferring cost of this nutrient enriched liquid swine manure to cultivable land with nutrient deficient soils increases (Møller et al, 2000), thus this manure is usually stored at the farms. The retention of nutrient rich manure slurry at the farms leads to environmental problems of groundwater pollution, surface water pollution (Zhang and Westerman, 1997; Steinfield et al., 2006), odour emissions, ammonia volatilization and greenhouse gas emissions. Cost reduction of hauling this manure slurry and nutrient value can be enhanced, by separating the manure slurry into a liquid part for farm utilization and the solid part loaded in nutrients and higher dry matter content which can easily be carried to farms having little livestock (Møller et al., 2000; Sørensen et al., 2003).

Separation of manure by chemical additives (coagulants, flocculants) have been studied before (Kayser, 1997; Henriksen et al., 1998a; Sømmer et al., 2005; Garcia et al., 2009). However, these studies used polymer additives, which requires very high residence time, storage in larger volume tanks and increased secondary toxicity effects on the environment. Thus, the combination of centrifugation and chemical additives to ameliorate separation of manure has been unsuccessful due to reasons of higher input costs, environmental problems and efficiency of the process.

Scientific studies done on centrifugation of swine manure with natural additive are lesser known until now. Biochar is a sustainable alternative and is a by-product of thermo chemical conversion (pyrolysis) of biomass. It has a good absorbent capacity, high porosity and high alkalinity (Joseph et al., 2009) by which it can bind with the manure particles and result into efficient sedimentation. Furthermore, biochar obtained from agricultural biomass has been proven effective in sorption of organic contaminants (Harris et al., 2009).

The RSREG procedure (Montgomery, 2009) uses mathematical and statistical techniques to analyse a problem in which a response is affected by many independent variables. Response surface analysis is done to determine the conditions optimum for the experiments.

This article discusses the results of experiments carried out to optimize the separation process for separating the low DM slurry into a high DM solid fraction. The study focuses on the parameters that affect the separation of liquid swine manure during centrifugation into a higher DM solid fraction. The main objective of this study was to develop an approach that would enable us to better understand the relationships between the variables (pH, biochar and centrifugation time) and the response (dry matter value after separation) and to obtain the optimum conditions for the separation of dry matter from liquid swine manure separation using central composite design (CCD) and response surface methodology (RSM).

Materials and Methods

Manure sampling

Raw swine manure was collected from a commercial farm in Québec with a dry matter content of 4% (w/v) and a pH of 7.2. The manure was stirred for a minimum of an hour using an electric stirrer, before being pumped directly from the pre-pit into a mobile tank. At the bottom of the mobile tank, an opening allowed the filling of 500 ml pre-weighed bottles. The manure in the temporary tank was agitated with an electric stirrer before transferring it to bottles. The bottles were transported to the laboratory on ice and maintained at 4°C in a cold chamber.

Centrifugation of samples

The raw manure slurry samples in the 500 ml bottles are homogenised by an agitator before centrifuging them in the centrifuge. Biochar is added in different percentage concentrations to the bottles of raw manure. pH is increased to the desired level by adding 1N NaOH solution and then the bottles are centrifuged at different time intervals. For obtaining the acidic pH (4.95) of 1N H_2SO_4 was added. During centrifugation of the samples the dense solid particles settle down in the bottles as precipitate and the lighter particles remain in the liquid fraction on top of the precipitate as the supernatant. The samples were centrifuged at maximum speed (Figure 1), equivalent to a RPM of 5500 for different time intervals to get the maximum dry matter separation in the separated solid fraction. The centrifuge used in laboratory is an IEC Model 2K centrifuge (International Equipment Company, 300 second avenue, Needham Heights, Massachusetts).

To know precisely the total mass of the liquid manure, each bottle was weighed with and without the liquid manure. The supernatant was then centrifuged under the effect of gravity. The remaining solid fraction (pellet) was weighed to determine the mass fraction separated.

The solid and liquid samples are then separated into different bottles and were analysed by a certified ISO laboratory. The DM content was determined at 105°C overnight or until consistent weight was achieved.

Figure 1: Laboratory centrifuge utilized for centrifugation of swine manure

Biochar

Biochar is a by product obtained by pyrolysis of solid fraction of swine manure and is being recycled in this process by using it as an additive. The biochar was obtained by pyrolysis reaction in a custom built pyrolysis reactor at *Centre de recherché industrielle, 333, rue Franquet, Québec* (Verma et al., 2010) from dried swine manure in batch over a temperature range between 200 to 600º C under vacuum. The pyrolytic vapour was condensed in two glass containers and the biochar was collected directly from the pyrolysis vessel after the completion of the pyrolysis batch.

The dosage of biochar was given in various percentages of 0.64%, 2%, 4%, 6%, and 7.36%.

pH determination

pH was changed by using 1N NaOH and 1N H_2S0_4 solution to get the desired pH level.

The pH meter used for measuring the pH was Denver instrument Ultra basic Bench top meters "Denver Instrument 5 Orville Dr., Bohemia, NY 11716".

Mass Balance

The mass balance followed for all the experimental runs in centrifugation of liquid swine manure into a solid and liquid fraction is given by the following equation for one experimental run:

$$Y = X_1 + X_2 \quad (1)$$

Where, Y is the initial mass (500 gm) of manure with 4% w/v dry matter

X_1 = Solid fraction (120 gm) separated with 26.3 % w/v dry matter

X_2 = Liquid fraction (380 gm) separated with 3.02 % w/v dry matter

Experimental Design

A three factor-five level Central Composite Design was utilized in this study and 20 experimental runs (Table 2) were performed (Montgomery, 2009). pH, biochar concentration and centrifugation time were the independent variables selected to optimize the separation of dry matter from liquid swine manure using centrifugation. The coded and uncoded levels of the independent variables are given in Table 1.

Table 1: Independent variables and coded factor levels for central composite design

Independent	**Coded Factor levels**				
Variables	**-1.682**	**-1**	**0**	**1**	**1.682**
pH (X_1)	4.95	7	10	13	14 (15.04*)
Biochar (X_2) (% w/v)	0.64%	2%	4%	6%	7.36%
Time (mins) (X_3)	9.55	30	60	90	110.4

Statistical Analysis

The experimental data obtained by following the above procedure was analyzed using RSREG procedure in SAS 9.2 (SAS Institute Inc., Cary, NC) by utilizing the following second-order polynomial equation:

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i \; x_i + \sum_{i=1}^{k} \beta_{ii} \; x_i^2 + \sum_i \sum_{\substack{j \\ i<j}} \beta_{ij} \; x_i x_j \qquad (2)$$

Where y is the response (Dry matter value, gm): x_i and x_j are the unicoded independent variables and β_0, β_i, β_{ii} and β_{ij} are intercept, linear, quadratic and interaction constant coefficients respectively. SAS 9.2 (SAS Institute Inc., Cary, NC) was used for analysis of variance (ANOVA) and for regression analysis.

Results and Discussion

Efficiency of dry matter separation by biochar

In this experiment, the dry matter separation obtained was maximum in a laboratory centrifuge until now with using a biochar and the dry matter value obtained with a maximum value of 33.06% w/v dry matter (Table 2) in solid fraction from raw swine manure having 4% w/v dry matter. (Chapman et al., 2004) studied that with a centrifuge decanter the maximum value obtained was 30.19 % w/v dry matter in solid fraction from liquid swine manure having 7.19% w/v dry matter. Whereas, in another study (Møller et al., 2007a) by a decanter centrifuge with a percentage dry matter of 5.1% of raw manure the percentage of dry matter obtained in the separated solid fraction was 32.7%.

Statistical analysis

The model was statistically tested by the Fisher's test for analysis of variance (ANOVA) (table 7). A very low probability value (0.0011) for total model by the Fisher's F-test indicated that the model was highly significant and the value of coefficient of determination (R^2) obtained was 0.8865 indicating that the variability of 88.65% in the response can be explained by the model.

The eigen values obtained by the analysis were all positive indicating that the stationery point was a minimum. The regression coefficients were calculated for the dry matter response and using the estimates in the model. The regression equation obtained is as follows:

$$Y = 45.50 - 3.50 \text{ pH} - 0.066 \text{ time} - 1.132 \text{ biochar} + 0.157 (\text{pH})^2 + 0.004 (\text{pH} * \text{Time}) + 0.003 (\text{time})^2 + 0.044 (\text{pH} * \text{biochar}) + 0.006 (\text{time} * \text{biochar}) + 0.160 (\text{biochar})^2 \qquad (3)$$

Table 2: Central composite design arrangement and responses of separated dry matter (%) values

Run	pH	Biochar	Time (mins)	pH	Biochar (% w/v)	Time (mins)	Dry Matter (% w/v) (Measured)	Dry Matter (% w/v) (Predicted)
1	-1	-1	-1	7	2	30	28.67	27.28
2	1	-1	-1	13	2	30	26.85	26.41
3	-1	1	-1	7	6	30	30.03	29.85
4	1	1	-1	13	6	30	30.71	30.04
5	-1	-1	1	7	2	90	28.82	28.32
6	1	-1	1	13	2	90	29.98	28.98
7	-1	1	1	7	6	90	33.06	32.33
8	1	1	1	13	6	90	33.84	34.06
9	-1.682	0	0	4.95	4	60	29.8	30.95
10	1.682	0	0	14	4	60	29.33	30.10
11	0	-1.682	0	10	0.636	60	24.5	25.90
12	0	1.682	0	10	7.364	60	32.1	32.33
13	0	0	-1.682	10	4	9.55	25.01	26.02
14	0	0	-1.682	10	4	110.4	29.64	30.26
15	0	0	0	10	4	60	27.82	27.30
16	0	0	0	10	4	60	26.87	27.30
17	0	0	0	10	4	60	26.04	27.30
18	0	0	0	10	4	60	27.75	27.30
19	0	0	0	10	4	60	27.82	27.30
20	0	0	0	10	4	60	28.11	27.30

The model equation indicates that there was significant effect of pH (X_1) on separated dry matter (Y) because it was having the largest coefficient. X_1(pH), X_2(centrifugation time), X_3(Biochar) having negative coefficients indicated linear effect to decrease Y. Among the linear, quadratic and cross-product forms of independent variables X_1($p<0.01$), X_1^2 ($p<0.01$) and X_3^2 ($p<0.05$) (Table 3) were significant. The maximum value of response obtained was pH of 13, centrifugation time of 90 minutes and biochar percentage of 6% with a dry matter percentage of 33.84 % w/v at the stationery point.

Table 3: Regression analysis of different parameters of dry matter separation in solid fraction from liquid swine manure

Independent Variable	Regression Coefficient	Standard Error	t Value	Pr > |t|
Intercept	45.506595	5.806862	7.84	<.0001 **
Linear				
pH (X_1)	-3.507869	0.848121	-4.14	0.0020 **
Time (X_2)	-0.064308	0.066538	-0.97	0.3566
Biochar (X_3)	-1.131189	0.997739	-1.13	0.2833
Quadratic				
pH*pH (X_1^2)	0.157292	0.038521	4.08	0.0022 **
Time*Time (X_2^2)	0.000330	0.000333	0.99	0.3443
Biochar*Biochar (X_3^2)	0.160638	0.074792	2.15	0.0573 *
Interaction				
Biochar*pH (X_{3*} X_1)	0.044167	0.067174	0.66	0.5257
Biochar*Time($X_{3*}X_2$)	0.006000	0.006717	0.89	0.3927
Time*pH (X_{2*} X_1)	0.004278	0.004478	0.96	0.3620

*Significant at 0.05 level; **Significant at 0.01 level.

Effect of various parameters on dry matter separation

The linear effect of biochar (additive) concentration (Table 3) had a negligible effect (p=0.2833) on increasing the separation of dry matter from the liquid swine manure. But, the quadratic effect (Table 3) had a significant effect (p<0.05) in increasing the separation of dry matter. The dry matter varied from 28.24 % w/v to 36.52 % w/v (Figure 2) at biochar concentration of 6% in centrifugation of the liquid swine manure and the maximum dry matter percentage obtained was 33.84 % w/v experimentally at pH of 13, percentage of biochar 6 % and centrifugation time of 90 minutes.

Centrifugation time (p > 0.1) had no significant effect (Table 3) on increasing the separation of dry matter from the liquid swine manure as the linear effect was negative.

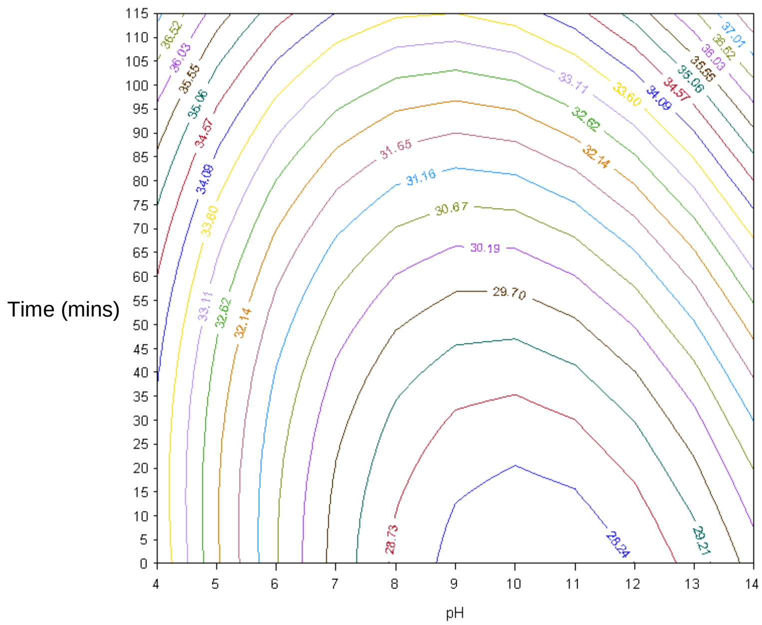

Figure 2: Contour plot of dry matter separation values at percentage of biochar 6%

Changing the pH of the liquid swine manure had a (Table 3) significant linear and quadratic effect on increasing the separation of dry matter from the liquid swine manure. At pH of 7 the maximum value obtained was 29.34 % w/v dry matter with a time of centrifugation 110 minutes and percentage biochar of 6%. At pH 13 (Figure 3), we can see from the contour plot that the percentage of dry matter increases to a value of 29.59 w/v at the time of centrifugation 110 minutes and percentage biochar of 6%.

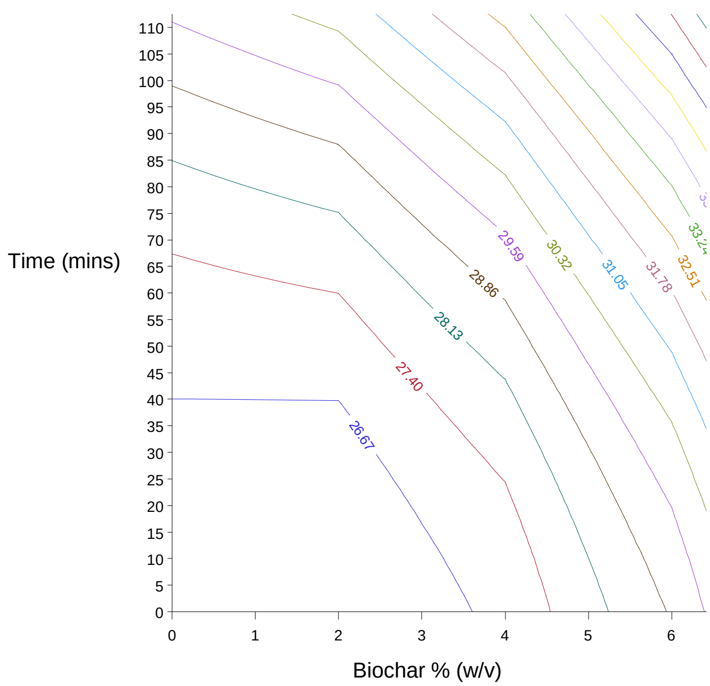

Figure 3: Contour plot of dry matter separation values at pH 13

Conclusions

Solid-liquid separation is an important part of manure management at the farm level. In this study we were able to separate liquid swine manure with 4 % w/v dry matter concentration with a maximum value of dry matter separation of 33.84% w/v at pH 13, time of centrifugation 90 minutes and biochar concentration of 6% and the minimum value of dry matter separation was 24.50 % w/v at pH 10, time of centrifugation 60 minutes, biochar concentration of 0.64 %.

The dry matter separation of 33.06 % w/v at centrifugation time of 90 minutes, percentage of biochar 6% and pH of 7 was an important value obtained at a similar pH of raw manure (pH =7.2). This percentage value of dry matter separation obtained at laboratory level was greater than value in the previous studies done on centrifugation of liquid swine manure (Chapman et al., 2004) with a decanter centrifuge which gave separation of 30.19 % w/v of dry matter from swine manure having 5.91 % w/v dry matter. Further study is recommended for getting better results with separation of dry matter with decanter centrifuge.

The Response surface model was significant with no lack of fit. The linear term of pH, quadratic terms of pH and biochar were significant. The other linear, quadratic and interaction terms were found to be non-significant.

Biochar used in this experiment is a byproduct of pyrolysis of swine manure which can be recycled in this process. With increase in dry matter separation the fraction of remaining liquid swine manure can be stored easily and transferred by pumping to remote areas. With increase in solid-liquid separation and better handling of manure at the farms will result in decrease in odour emissions to the environment and prevent leaching of manure to the groundwater.

Further studies need to be done for increasing the dry matter separation by doing particle size studies on biochar and doing separation by decanter at field level.

Acknowledgements

Financial support for this study was provided by the *Ministère de l'Agriculture, des Pêcheries et de l'Alimentation du Québec* (MAPAQ). The authors are thankful to Research and development Institute for Agri-Environment, Québec for their technical support and contribution in conducting this study. Finally, we thank Michèle Grenier for helping us in doing the data analysis.

References

Chapman A.M., 2004. Séparation du lisier de porc par centrifugation : Evaluation et amélioration des performances. MS thesis, Québec. Université Laval, Canada, Département des sols et de génie agroalimentaire.

Douglas C. Montgomery, 2009. *Response Surface Methodology: Process and Product Optimization Using Designed Experiments*. 3rd Edition, Wiley eastern.

Drolet. J. Y. and Gilbert. D., 2000. "Le portrait agroenvironnemental des fermes du Québec: Rapport sectoriel: Porcs." *Rep. No. 179 PROD73*. Union des producteurs agricoles. *Ministère de l'agriculture des pêcheries et de l'alimentation du Québec*. Longueil. Canada.

Garcia M.C., Szogi A. A., Vanotti, M.B., Chastain J.P., Millner P.D. 2009. Enhanced solid–liquid separation of dairy manure with natural flocculants, *Bioresource Technol*. DOI: 10.1016/j.biortech.2008.11.012.

Henriksen K., Berthelsen L., Matzen R. 1998a. Separation of liquid pig manure by flocculation and ion exchange. Part 1. Laboratory experiments, *J. Agr. Eng. Res*. 69, 115–125.

Kayser R., 1997. Technologische bewertung der FuE: Vorhaben in: KTBL (Eds.). Statusseminar “Umweltverträgliche Gülleaufbereitung und -verwertung”: Congress Proceedings. Arbeitspapier Nr. 242. Münster Hiltrup. Germany. pp. 2 24–30.

Joseph Stephen and Lehmann Johannes, 2009. *Biochar for Environmental Management Science and Technology*.London, U.K.: Earthscan.

Møller H.B., I.Lund and S.G. Sommer., 2000. Solid-liquid separation of livestock slurry: efficiency and cost. *Bioresour. Technol.* 74:223-229.

Møller H.B., Hansen J.D., Sørensen C.A.G. 2007a. Nutrient recovery by solid-liquid separation and methane productivity of solids, *Trans. ASABE* 50, 193–200.

SAS., 2010 *SAS User's Guide: Statistics*. Ver. 9.2. Cary, N.C.: SAS Institute, Inc.

Sørensen C.G., Jacobsen B.H. and Sømmer S.G., 2003. An assessment tool applied to manure management systems using innovative technologies. *Biosyst. Eng.* 86: 315–325.

Sømmer S.G., Mathanpal G. and Dass T.T., 2005. A simple biofilter for treatment of pig slurry in Malaysia. *Environ. Technol.* 26: 303–312.

Steinfeld H., Gerber P., Wassenaar T., Castel V., Rosales M., de Haan C., 2006. Livestock’s Long Shadow: Environmental Issues and Options, FAO, Rome.

Verma M, Godbout S, Larouche J.P., Lemay S.P., Pelletier F, Solomatnikova O, 2010. Vacuum pyrolysis of swine manure: biochar production and characteristics, XVIIth World Congress of the International Commission of Agricultural Engineering (CIGR), June 13-17 2010. CSBE: 101374.

Willie Harris., Lena Ma, Bin Gao and Xinde Cao, 2009. Dairy-manure derived biochar effectively sorbs lead and atrazine. *Environ. Sci. Technol*.: 43.3285-3291.

Zhang R.H. and P.W. Westerman., 1997. Solid-liquid separation of annual manure for odor control and nutrient management. *Applied Eng. in Agric.* 13(2): 383-393.