Treatment of Faecal Sludge by Two Biochemical Processes

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Abstract: Untreated raw faecal sludge is generally reused as fertilizer by some farmers and market gardeners to improve their production areas. However, untreated sludge contains pathogenic germs which, via the faecal-oral route, can present a microbial risk for consumers of fertilized products. To reduce this risk, the objective of this work was to evaluate the effectiveness of their hygienization by two chemical processes: the use of urea (H_2CONH_2) and ammonium sulphate (($NH_4)_2SO_4$). The results obtained showed that the addition of 2% (w/w) of urea in the sludge increased the pH around 9 and was sufficient to increase the inactivation of the pathogenic germs sought (Thermotolerant coliforms, Escherichia coli, Fecal streptococci, Sulphite-reducer anaerobes). The ammonium sulphate amendment had no appreciable effect on the reduction of the concentrations of these microbiological germs. However, treatment with these two additives reduced the levels of metallic trace elements (Zn, Cu, Ni, Pb, Cd and Cr) in the treated sludge. Then, the settling test made it possible to optimize the drying time of the sludge treated with sludge index values less than 100 mLg^{-1}. Urea treatment is therefore a simple and reliable approach to obtain hygienic and agronomically ready sludge. Subsequent work will concern agronomic trials of treated sludge.

Keywords: Faecal Sludge, Urea, Ammonium Sulphate, Inactivation of Pathogenic Germs, Agronomic Uses

1. Introduction

Reusing faecal sludge in agriculture as a fertilizer has many potential benefits from the fertilizing properties of the nutrients present, but it also poses risks to human health [1, 2]. Faecal sludge contains large amounts of pathogens such as bacteria, helminths, viruses, protozoa and other contaminants such as trace metal elements and organic substances [3, 4]. In addition, they are highly fermentable and are at the origin of the production of gases (amino acids, hydrogen sulphide, mercaptans) that cause olfactory nuisances and disease vectors [5]. Thus, by reusing them as agricultural fertilizers without proper treatment, they can end up harming the environment and affecting public health [6]. Some farmers and especially market gardeners use raw sludge as a soil amendment. However, many pathogens present in these sludge can present a microbial risk to consumers of fertilized products via the faecal-oral route. Thus, by sanitizing the sludge, all successive sludge management activities can be carried out safely [7].

To limit the process of rapid biodegradation of these sludge drains, it is possible to develop on the treatment lines, a pre-treatment step by stabilization aimed at obtaining sludge that no longer evolves or, at the very least, evolves slowly, both biologically and physico-chemically [8, 9].

In this study, two interesting and innovative treatment technologies were studied in particular the biochemical treatment of faecal sludge by urea (H_2CONH_2) and ammonium sulphate ($(NH_4)_2SO_4$) which are viable options for effective inactivation of pathogenic microorganisms and reduction of their volume through optimization of settling time.

The objective of this work was therefore to evaluate the effectiveness of the hygienization of the sludge by these two biochemical processes. Indeed, it is a question of reducing the fermentable power of the sludge of emptying by oxidizing the organic matter so as to make it more easily bio-assimilable, by inactivating the reducing germs responsible for fermentation and by reducing the concentration of metallic pollutants. Thus, the treated sludge is hygienic,

deodorized and does not obviate agricultural devolution which remains the preferred destination.

2. Materials and Methods

2.1. Treatment

Sludge hygiene, using urea and ammonium sulphate, was estimated at laboratory temperature, which averaged 27°C. Both additives were added at concentrations of 0%, 1%, 2% and 3% (w/w). Treatment began a few hours after the collection of the faecal sludge. A sample without the two additives (0 % w/w) was the reference for physicochemical and microbiological treatment. The urea used for the treatments came from a local agricultural store and was 95% pure. With regard to ammonium sulphate, it came from the chemistry research laboratory of the University of Kara with a purity of 99.5%. However, for the urea and ammonium sulphate dose, the measured sludge density was 1.07g.mL^{-1} so that the equivalence of 1 kg = 1 L was applied. Thus, for the addition of urea and ammonium sulphate, the calculation was made as indicated in Equation 1.

$$D_{ad} = \frac{m_{FS}(g) \times C_{ad}(\%)}{P_{ad}(\%)} \qquad (1)$$

With,
D_{ad} = dose of additive (urea or ammonium sulphate);
m_{FS} = mass of faecal sludge;
C_{ad} = concentration of the additive;
P_{ad} = purity of the additive.

For this study, seven (07) 500 mL glass vials were used as reactors for sludge treatment, of which 3 were for each additive and 1 were additive-free (reference). The assembly was then placed on the laboratory bench (Figure 1).

Figure 1. *Treatment reactors: a. urea; b. no additive; c. ammonium sulphate.*

After adding granules of each additive, the sludge was mixed manually with a glass rod for about 5 minutes. Then the reactors are sealed for further reactions.

The treatment lasted 7 days. After treatment, the treated sludge was dried with a 90% drying rate and stored for future agronomic tests.

The dry matter (DM) content was assessed by equation 2. In fact, a mass m$_1$ of sludge is dried in the oven at 105°C, until a constant mass m$_2$ is obtained after 24 hours.

$$DM = \frac{m_2 \times 100}{m_1} \qquad (2)$$

2.2. PH Measurements

The pH was checked with a pH meter electrode, Storius PT-10, before and after the addition of the two additives (urea and ammonium sulphate). Then, additional pH tests were performed daily during the seven (7) days of treatment to verify if the addition of these additives changes the pH of the fecal matter. However, the reactors were mixed manually for 1 minute before pH measurements.

2.3. Organic Matter Content

The organic matter was determined by the fire loss method. It was used to directly measure the organic matter in the treated sludge. The samples were dried for 16 hours in an oven at 105°C. Thus, a mass of each treated faecal sludge was taken from a dry crucible. Then, the crucible and its contents are placed in an oven for 16 hours at a temperature of 375°C [10]. The percentage of organic matter is given by Equation 3.

$$\%OM = \frac{m_1 - m_2}{m_1 - m_0} \times 100 \qquad (3)$$

where
OM: organic matter (%);
m_0: mass of the empty crucible (g);
m_1: final mass (g);
m_2: mass of the crucible containing the ash (g).

2.4. Sludge Volume Index

The sludge volume index (SVI) test is used to assess the suitability of sludge hygienic by these two processes for settling.

A 25 mL of hygienic sludge collected from a 100 mL test tube is added 75 mL of clarified water to induce ¼ dilution [9]. After some stirring by a movement from the bottom to the top of the hermetically closed specimen, the mixture is left at rest at the temperature of the laboratory and on a horizontal bench isolated from all vibrations. After 30 minutes of settling, the level of the sludge volume in the test tube is read and noted VS$_{30}$ (Figures 2a and 2b).
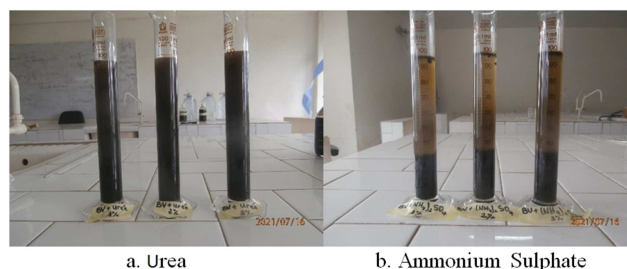

Figure 2. *Settling test in the test tube, a. sludge treated with urea; b. those treated with ammonium sulphate.*

The sludge index represents the volume occupied by one gram of sludge after thirty minutes of static settling in a one-litre cylinder with a graduated transparent wall [11]. The index measured is denoted SVI, expressed in mL.g^{-1} of SS, and is defined by equation 4.

$$SVI = \frac{VS_{30}}{SS} \times 100 \qquad (4)$$

with:

VS_{30} = volume of sludge settled in thirty minutes (mL.L^{-1}).
SS = concentration of suspended solids in the test specimen (g.L^{-1}).

The determination of SS is made by filtration on glass fiber filter taking into account the domestic origin of the effluents. The measurement of SS by filtration is based on the principle of double weighing: a volume of sludge is filtered on a membrane (previously weighed empty) of 1.5 microns and the residues on the latter are weighed [12].

2.5. Trace Metal Content

The samples are mineralized by acid digestion in water. Each sample is made up of 3 test pieces of 1 g. Each sample is placed in a glass Erlenmeyer where it receives 3 mL of distilled water, 7.5 mL of hydrochloric acid (38%, normapur) and 2.5 mL of nitric acid 65%, normapur). The mixture is sealed and left at room temperature for 12 hours. The mineral is then concentrated by boiling for 2 hours. After cooling, the volume is adjusted to 20 mL with distilled water Mineralized blanks (without sludge) are prepared simultaneously [9, 13]. The determination of metallic trace elements in minerisâts is carried out by flame-mode atomic absorption spectrometry (Aanalyst 800/PERKIN ELMER).

2.6. Microbiological Analysis

Determination of Thermotolerant Coliforms, Escherichia coli, Fecal Streptococci, Anaerobic Sulfito-Reducer Agents, Salmonella sp and Parasites (Entamoeba coli cysts) in Crude Sludge and stabilized sludge is based on research and enumeration of bacterial colonies by membrane filtration technique and seeding in appropriate culture media followed by incubation at the proper temperature for 18 to 24 hours. The analyses were carried out according to the methods of routine analysis of the French Association for Standardization (AFNOR): decree of 21 December 1979.

3. Results and Discussion

3.1. Effects of Additives

3.1.1. Effects of Urea

Urea, when added to faecal sludge, is catalyzed by the urease enzyme, which is present in feces, and breaks down into ammonia and carbon dioxide. The decomposition of urea results in an alkaline pH that affects the balance between ammonia and ammonium, thus promoting the formation of ammonia. The addition of 1%, 2% and 3% (w/w) urea resulted in an increase in pH to about 8.50 in 3 days, based on urea hydrolysis, so the pH rise was observed in treatments (Figure 3 and Table 1). However, a slight decrease in pH can be seen when urea is added immediately before the alkalinity of the reaction medium is observed.

Indeed, the acidity observed at the beginning could be explained by the rapid evolution of nitrogen in nitric form NO_3^-. However, the pH recorded in untreated sludge (0%) ranged from 7.70 to 7.73.
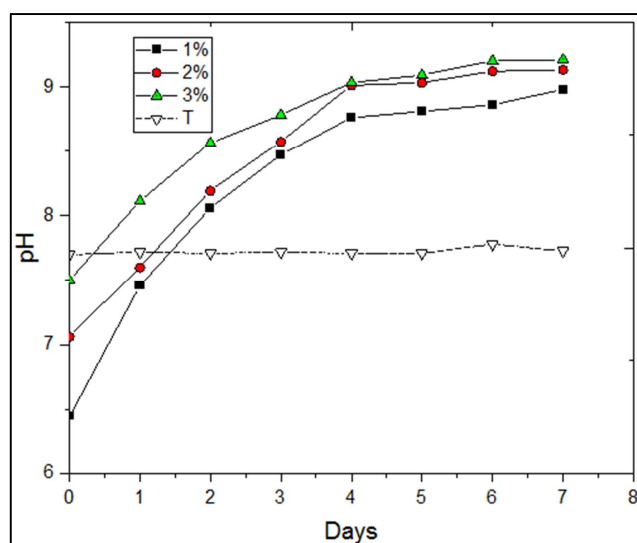

Figure 3. PH recorded during treatments.

Table 1. Minimum values and maximum recorded.

Sludge treatment	Measured pH	
	Min	Max
3%	7,50	9,21
2%	7,05	9,13
1%	6,44	8,98
0%	7,70	7,73

Non-ionized ammonia (NH_3) acts as the main hygienic agent. Inactivation of pathogens by non-ionized ammonia is observed for several types of microorganisms, bacteria, viruses and parasites [7].

Urea is rapidly hydrolyzed in the volatile form, ammonia (NH_3), in a reaction catalyzed by urease enzymes (urea amidohydrolase). Carbamate (H_2N-COOH) produced in this reaction then spontaneously hydrolyses to carbonic acid (H_2CO_3) and releases a second molecule of NH_3 [14] (Equation 5).

$$H_2 - CO - NH_2 \xrightarrow{\ Urease\ } H_2N - COOH + NH_3$$

$$\xrightarrow{\ H_2O\ } H_2CO_3 + 2NH_3 \qquad (5)$$
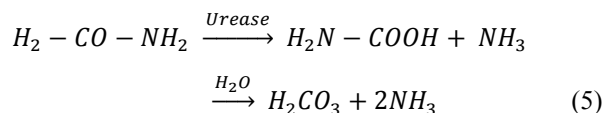
The likely bacterial inactivation mechanisms of NH_3 would be protein denaturation, membrane destruction, or rapid cytoplasm alkalization, resulting in critical potassium (K) loss. Viral inactivation is likely due to RNA cleavage. Thus, the results of the microbiological analyses have shown that the higher the rate of urea, the more the germs sought are destroyed with very high rates of abatement. But the search for salmonella sp and Entamoeba cysts is negative (Table 2).

Table 2. *Effects of urea on microbiological germs.*

| Germes	UFC/g			
	T	FS+ 1%U	FS+ 2%U	FS+ 3%U
Thermotolerant coliforms (44°C)	5 000	60	30	8
Escherichia coli	2 200	40	20	6
Streptocoques fécaux (37°C)	18 000	4100	100	30
Sulfite-reducer anaerobes (44°C)	140 000	320	180	50
Salmonella sp	-	-	-	-
Entamoeba cysts coli	-	-	-	-

T= FS+0%U; FS: faecal sludge; U: urea

3.1.2. Effects of Ammonium Sulphate

Ammonium sulphate, $(NH_4)_2SO_4$, added to the sludge reduced the pH of the reaction medium. Thus, the higher the level of the additive, the lower the pH becomes (Figure 4 and Table 3). Acidification is due to the addition of SO_4^{2-} ions and the evolution of nitrogen in the nitric form NO_3^- in the sludge (Equation 6).

$$(NH_4)_2SO_4(s) + H_2O\ (l) \rightarrow 2NH_4^+(aq) + SO_4^{2-}(aq) \quad (6)$$
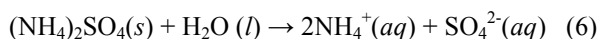
Indeed, the nitric form NO_3^- is obtained by the biological elimination of ammonium ions NH_4^+, nitrification, is traditionally defined as the aerobic oxidation of ammonium ion to nitrate through nitrite ion [15]. This oxidation is carried out by two groups of ammonium oxidizing autotrophic bacteria represented by the genera Nitrosomonas and Nitrosococcus which oxidize ammonium ions to nitrites (NO_2^-) (Equation 7); and nitrites represented by the genera Nitrobacter and Nitrospira which oxidize nitrites to nitrates (NO_3^-) (Equation 8).

$$NH_4^+ + \frac{3}{2}O_2 \rightarrow NO_2^- + 2H^+ + H_2O \quad (7)$$

$$NO_2^- + \frac{1}{2}O_2 \rightarrow NO_3^- \quad (8)$$
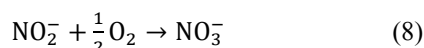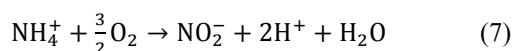
Table 3. *Minimum values and maximum recorded.*

Sludge treatment	Measured pH	
	Min	Max
3%	3,47	7,08
2%	3,38	7,07
1%	5,45	7,08
0%	7,70	7,73

However, the pH of the treated sludge rose rapidly and stabilized around 7 after 24 hours (Figure 4 and Table 3). The small rise in the pH of the reaction medium could be justified by the transformation of part of NH_4^+ into NH_3 (Equation 9).

$$NH_4^+(aq)+ H_2O(l) \leftrightharpoons NH_3(aq) + H_3O^+(aq) \quad (9)$$
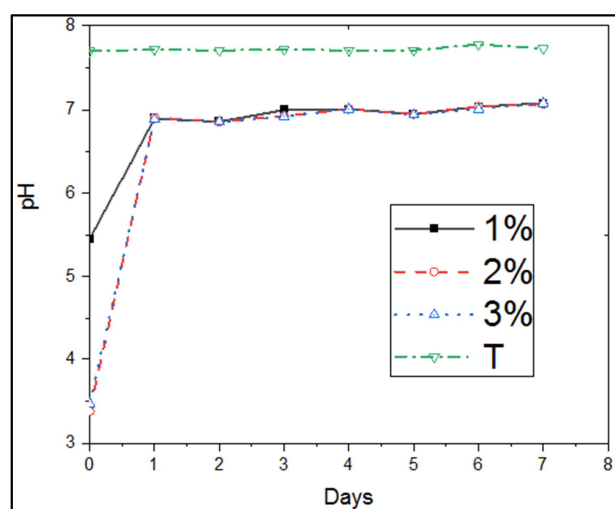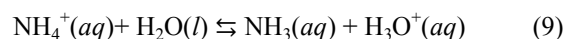
Figure 4. *PH recorded during treatments.*

The results of the microbiological analyses (Table 4) show that there were destruction effects of the germs sought in the study with average abatement rates. The sudden drop in pH from 7.73 to 3 and 5 made the medium acidic. The acidity of the reaction medium would be responsible for the effect of this destruction of these germs and also for the suppression of olfactory nuisances (H_2S, mercaptans..) and inhibition of fermentative activity [9].

Table 4. *Effects of ammonium sulphate on microbiological germs.*

Germs	UFC/g			
	T	FS+1%AS	FS+ 2%AS	FS+ 3%AS
Thermotolerant coliforms (44°C)	5 000	1000	600	500
Escherichia coli	2 200	2100	2000	2000
Streptocoques fécaux (37°C)	18 000	17000	15000	14000
sulfite-reducer anaerobes (44°C)	240 000	24000	20000	18000
Salmonella sp	-	-	-	-
Entamoeba cysts coli	-	-	-	-

T= FS+0%AS; FS: faecal sludge; AS: ammonium sulphate.

***Table 5.** Levels of trace metal elements.*

Samples	mg.kg^{-1}					
	Cd	Pb	Cr	Cu	Zn	Ni
FS+ 0%U	1,10 ± 0,24	14,00 ± 1,25	27,50 ± 2,44	62,50 ± 1,88	471,51 ± 5,24	12,50 ± 0,09
FS+ 1%U	0,92 ± 0,11	5,01 ± 0,48	18,50 ± 1,48	40,23 ± 2,42	358,50 ± 1,49	11,21 ± 1,32
FS+ 2%U	0,85 ± 0,06	3,08 ± 0,23	14,50 ± 0,87	31,15 ± 1,02	234,02 ± 2,74	11,05 ± 1,05
FS+ 3%U	0,78 ± 0,15	0,55 ± 0,17	13,09 ± 0,18	29,65 ± 0,87	209,97 ± 2,01	10,59 ± 1,65
FS+1%AS	1,06 ± 0,05	9,50 ± 0,09	13,52 ± 0,24	47,01 ± 1,75	421,03 ± 2,35	11,51 ± 0,45
FS+2%AS	0,98 ± 0,08	6,42 ± 0,34	12,70 ± 0,04	38,25 ± 2,58	398,46 ± 3,66	9,19 ± 0,13
FS+3%AS	0,90 ± 0,18	4,50 ± 0,66	11,85 ± 0,51	35,57 ± 1,09	354,78 ± 1,84	3,05 ± 0,44

FS: faecal sludge; U: urea; AS: ammonium sulphate.

3.2. Effects on Metallic Trace Elements

The results recorded in Table 5 suggest that there have been surface complexions. Indeed, in the reaction medium there is an anionic group that functions as inorganic ligands (OH$^-$, Cl$^-$, SO$_4^-$) and that can react with metal ions. Thus, these surface sites form chemical bonds with the ions in solution [16].

The alkaline urea keeps the treated sludge at pH 9 to 10, thus minimizing the solubility of the metals found in it by complexation or precipitation [9]. Often, for a very basic medium (pH 9) complex ions can contain up to six Pb atoms (for example [Pb$_6$O(OH)$_6$]$_4^+$) [16].

The addition of ammonium sulphate releases sulphate ions. Thus, these sulphate ions precipitate some of these metal ions. However, some precipitates are insoluble in aqueous medium. It can be seen that the higher the concentration of ammonium sulphate in the reaction medium, the greater the concentration of the element Pb. The decrease in Pb content could be explained by the very stable form of lead sulphate (II) precipitate (PbSO$_4$) in the aqueous medium.

3.3. Organic Matter Content

Organic matter greatly amplifies the cationic exchange capacity of the soil and retains nutrients available to plants [9]. Thus, the determination of organic matter (OM) levels allowed an assessment to be made of the proportion of organic matter present in BVs processed in these two biochemical processes compared to that of raw faecal sludge. The results of the organic matter contents are 69.24 ± 0.71, 70.71 ± 0.48 and 73.91 ± 0.32% respectively for the hygienic faecal sludge by the addition of 1%, 2% and 3% urea. But these contents are 70.31 ± 0.9, 81.37 ± 1.02 and 83.81 ± 0.84% respectively for faecal sludge treated with 1%, 2% and 3% ammonium sulphate additions (Figures 5a and 5b).
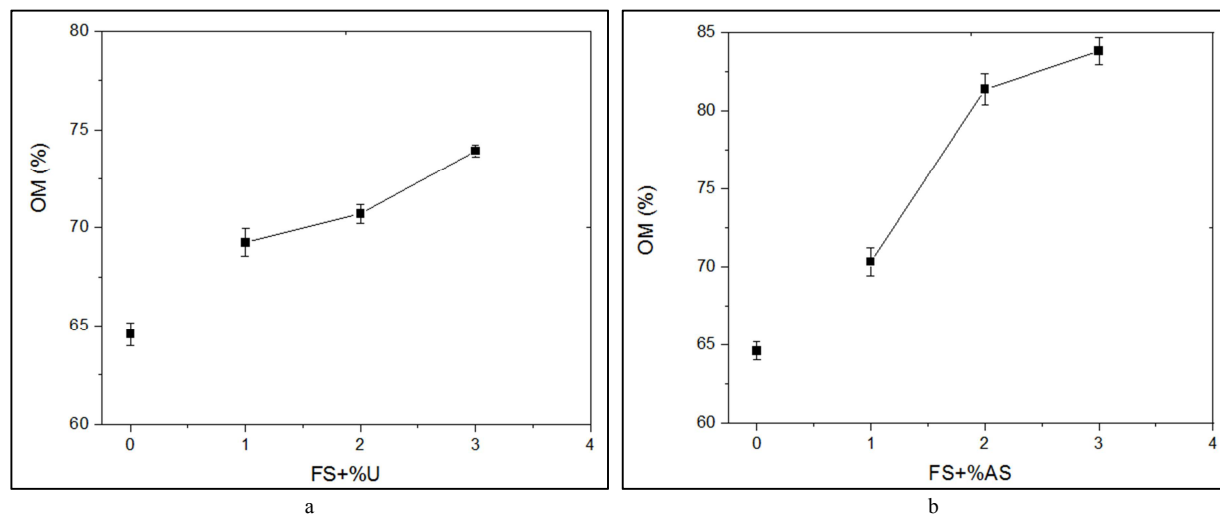
***Figure 5.** Percentage of organic matter: a. urea-treated sludge; b. ammonium sulphate-treated sludge.*

But the organic matter content is 64.58 ± 0.58% in untreated faecal sludge. The results in Figure 4 show that it is the addition of urea or ammonium sulphate, the more they increase, the greater the percentage of organic matter in the treated sludge. The increase in organic matter levels in treated sludge can be explained by the inputs of organic nitrogen from both additives. In addition to the percentage of organic matter in the faecal sludge treated with ammonium sulphate, percentages of organic matter in urea-hygienic faecal sludge are found to be relatively low. This difference could be due to the fact that urea reacted more. So there is a slight loss of dry matter by urea during reactions. However, both additives have very beneficial effects on their organic matter intake.

3.4. Settling Test

The sludge index (SI) is an indispensable tool for the

operator of a fecal sludge drying bed station to characterize sludge for dehydration [9]. For this treatment process, the hygienic sludge index calculations give 35.00 ± 0.12, 34.00 ± 0.21 and 34 ± 0.10 mL.g^{-1}, respectively, for faecal sludge treated with 1%, 2% and 3% ammonium sulphate. For the treatment of sludge with urea, the sludge index results give by 43.00 ± 0.34, 42.00 ± 0.16 and 41 ± 0.43 mL.g^{-1} respectively for additions of 1%, 2% and 3% of urea. Gold for the lower sludge index 100 mL.g^{-1} faecal sludge sediments easily and are most often well mineralized (DVS < 65%) [17]. Thus, these sewage sludge treated with urea and ammonium sulphate are suitable for treatment by settling and drying [9]. However, by parallelism of the sludge indices, it can be seen that the sludge treated with ammonium sulphate is more suitable for settling because of the addition of ferric chloride (FeCl$_3$). It is noted that the addition of FeCl$_3$ to the mixture of faecal sludge + urea and to that of faecal sludge + ammonium sulphate favored the advanced dehydration of hygienic sludge by the formation of amorphous gels [9, 18, 19]. Thus, faecal sludge treated by the sulphate process optimized the drying time once allowed in the drying beds. All in all, both processes have economic interests in settling.

4. Conclusion

Research into the hygienic upgrading of faecal sludge has led to two innovative biochemical treatment processes. Thus, the addition of 1%, 2% and 3% (w/w) of urea in the sludge at an average temperature of 27°C is sufficient to produce a safe sludge. *Thermotolerant Coliforms, Escherichia coli, Faecal Streptococci and Sulfito-reducing Anaerobes* were inactivated by increased ammonia concentration making the reaction medium more alkaline with a pH around 9. Under the same conditions, the addition of ammonium sulphate caused the pH of the sludge to drop around 7, which did not have any significant inactivation effects of the pathogenic germs sought. However, the decrease in pH has led to an increase in the concentration of NH$_4^+$ which will bring a fertilizing effect of the treated sludge. In addition, the application of the two precedents reduced the concentrations of trace metal elements (Zn, Cu, Ni, Pb, Cd and Cr). Then, the settling test applied to these treated faecal sludge assessed their suitability for settling by their sludge indices ranging from 34.00 ± 0.10 to 43.00 ± 0.34 mL.g^{-1}. With sludge index values well below 100 mL.L^{-1} these treatment processes will increase the dryness of the sludge and reduce the drying time of these treated sludge once admitted into the drying beds.

Approaches are shown to be effective, with short periods of remediation and simple technology. Apart from the disinfectant effects, the treatment with urea or ammonium sulphate increased the nutrient and fertilizing values of the sludge for reuse in agriculture. However, disinfection of faecal sludge with large-scale urea and its application in case of emergency is more efficient. Subsequently, the hygienized faecal sludge will be tested by agronomic trials for their fertilizing effects.

Acknowledgements

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