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## Use of silica fume and nano-silica in mortars attacked by acids present in pig manure

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### Abstract

Pig houses provide a highly aggressive environment to concrete structures. The chemical aggressiveness generated by pig manure may cause wear, loss in strength and premature damage to floors, leading to animal hoof diseases. Since the chemical attack in cementitious structures is controlled by its porosity and permeability, the use of mineral admixtures in cement is convenient. This work evaluated the behaviour of cement mortars with nano-silica and silica fume (micro-silica) to an acid attack, simulating the effects of acids present in pig manure to cementitious materials. Two cement mortars were tested: one with nano-silica as an additive and another with nano-silica and silica fume (cement replacement of 10%), as well as a control mortar with Portland cement. The mortars were submitted to acid attack cycles for four weeks. The compressive strength at 35, 50 and 65 days, mass loss and water absorption after 65 days were evaluated. Mortars containing nano-silica and silica fume showed considerable loss of workability, however showed the best performance in all studied aspects, indicating that the simultaneous use of these additions may result in longer-lasting concrete structures in pig houses.

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## 1. Introduction

Brazil is among the world top four pork producers and exporters, along with China, the European Union and the United States (ABPA (2017)). The importance of the design and durability of the structures used in pig houses is well established (De Belie et al. (2000)) and the issue needs further studies in Brazil, since the productivity could be affected. Concrete and mortar are the most frequently used materials in farm buildings (Bertron et al. (2004)), and in pig houses, they are found in concrete slatted and solid floors, storage concrete manure tanks, mortar-coatings and silos. However, livestock media is aggressive to cementitious materials (Massana et al. (2013)).

One of the main concerns is about degraded concrete floors, which have been directly related to hoof disorders, leading to lameness and economic losses due to reduced performance and longevity (Olsson et al. (2016)). Rough surfaces cause grazes and premature wear of animal hooves. Furthermore, affected floors are hard to clean, promoting diseases (De Belie et al. (2000)).

The Brazilian standards ABNT-NBR 6118:2014 and ABNT-NBR 12655:2015 set out minimum concrete parameters depending on the concentration of chlorides and sulphates in the environment, and the use of sulphate-resistant cement is mandatory in case of highly aggressive media. However, in Brazil, there is not a specific recommendation for cement or concrete used in agricultural structures and the many types of aggressiveness farm buildings might have. Several countries developed national standards or guidelines, setting parameters and minimum standards for this type of structure. Concrete made with sulphate-resistant cement and cement with fly ash are recommended for agricultural structures in Spain (Svennerstedt et al. (1999)). The Irish government sets out the minimum concrete specification for use in agricultural structures and mentions the use of ground granulated blast-furnace slag or silica fume (Ireland (2017)).

### 1.1. Pig manure and its aggressiveness to cementitious materials

Pig manure has a very heterogeneous composition (Kunz et al. (2009)), influenced by factors such as age, physiology, breed and method of farming (Massana et al. (2013)), leading to variable pH values, ranging from 5.30 (Zhang H. et al. (1994)) to 7.72 (Tavares (2012)). Although its pH cannot be considered aggressive to concrete, the presence of organic acids along with sulphate salts might be (De Belie (2000)).

The short chain organic acids (e.g acetic, propionic and butyric acid) are formed naturally by biological action, characterized by the acidogenesis of the organic matter present in manure. Bortoli (2014) evaluated the kinetics of generation-consumption of these acids during the storage of swine manure and found concentration peaks in the range of 2.3 to 2.7 g/L.

Large amounts of lactic and acetic acid as well as the aggressive ions  $\text{Cl}^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{Mg}^{2+}$  and  $\text{NH}_4^+$  have been observed on floors of pig houses (De Belie et al. (2000)). Organic acids are very aggressive since they can combine with free lime ( $\text{Ca}(\text{OH})_2$ ) present in cementitious materials producing very soluble calcium salts (De Belie (1997)). The leaching of calcium increases the paste porosity, helping the entrance of other agents such as high concentrations of  $\text{CO}_2$  from animals' respiration, causing carbonation. Ammonia gases and  $\text{H}_2\text{S}$  from the manure could lead to sulphate attack (Svennerstedt et al. (1999)). Moreover, these aggressive agents tend to decrease concrete pH (from around 13 to around 9), leading to the corrosion of reinforcing steel bars, either by carbonation or chloride attack. Corrosion reduces the cross-sectional area of the steel bars, and its products, which have a larger volume than the steel itself, induce tensile stress in concrete, resulting in cracking (Song H.W. and Saraswathy V. (2006)) and eventual breakage of slatted floors.

The leaching of the cement paste also causes the exposure of aggregates, increasing the floor roughness. Due to its mechanical impact, the use of high-pressure water in the cleaning process may also influence the floor degradation (De Belie et al. (2000)).

Besides water/cement ratio and cement content, the cement type, pozzolanic additions, aggregate type, polymer additions, application of cement bound surface layers as well as impregnation with water repellents could influence concrete degradation by lactic and acetic acid (De Belie et al. (2000)). In addition, the floor design should be considered (solid or slatted, the beam shape and gap width) (Ye et al. (2007), Yazici and Inan (2006)).

## 1.2. Use of silica fume (SF) and nano-silica (NS) as an addition

Silica fume (SF) is a by-product of the silicon and ferrosilicon alloy production and consists of spherical particles (0.1-0.2  $\mu\text{m}$ ) with high contents of amorphous  $\text{SiO}_2$  (85-90%) (Taylor (1990)). That makes silica fume a highly reactive pozzolan, other than providing an effective filling effect. The pozzolan reactions reduce the content of  $\text{Ca}(\text{OH})_2$ , refine the pore structure, and decrease permeability as a result. Pore size is probably the most important factor controlling the deterioration rate of concretes submitted to aggressive media (Mehta (2006)).

Various authors have reported pore refinement and lower permeability with the incorporation of SF in cementitious materials (Dewiler and Mehta (1989), Hoffman (2001), Kulakowski (2002), Chung (2002)). According to De Belie et al. (1997), the concrete degradation in a liquid containing lactic and acetic acid was nearly halved with the addition of SF when compared to the concrete of reference. Other pozzolans may have similar effects such as metakaolin and rice husk ash (De Belie et al. (2000)). The use of sulphate-resistant cement may also be a viable solution to increase durability of concretes submitted to pig manure (Abdelmesh (2008)), since it has lower  $\text{C}_3\text{A}$  content, decreasing sulphate attack.

More recently, many studies have been conducted about the incorporation of nano-silica (NS) in cementitious materials. Authors report increase in compressive strength, pore refinement, and an improved C-S-H (Hou et al. (2013), Soares (2014), Neto et al. (2017)). A more solid and homogenous C-S-H would increase the leaching resilience of calcium and increase durability (Gaitero et al. (2008)). The use of 3.8% of nano-silica in cement would elevate the compressibility, tensile strength, and concrete durability (Quercia G et al. (2012)). The use of NS in conjunction with SF has shown even better results (Senff et al. (2010b), Nili et al. (2010), Hendi et al. (2017), Mendes et al. (2017), Li et al. (2017)).

## 2. Experimental Process

This study aimed at evaluating the behaviour of two cement mortars: one with nano-silica and another with nano-silica and silica fume, as well as a control mortar with Portland cement. The mortars were immersed in an aggressive solution, in order to simulate the effects of acids present in pig manure to cementitious materials. The loss of compressive strength, loss of mass and water absorption were evaluated throughout the test.

### 2.1. Materials

Three types of mortar were produced with Portland cement, namely CPV-ARI, according to ABNT-NBR 5733:1991. Control mortars were made with superfluidifying polycarboxilate additive (PA); NS mortars contained a superfluidifying polycarboxilate additive modified with stabilised nano-silica and NS+SF mortars were made with silica fume and nano-silica additive. The mix proportions in mass of the three types of mortar are presented in Table 1.

Table 1. Mix proportions of mortars.

Type	Cement	Sand	W/C	SF	NS	PA
Control	1	3	0.5	-	-	1%
NS	1	3	0.5	-	1%	-
NS+SF	0.9	3	0.5	0.1	1%	-

After mixing, the flow table test (ABNT-NBR 7215:1996) was performed with the three types of mortar. The hardened cement mortars were cylindrical, 100 mm high and 50 mm in diameter. The specimens were demoulded 24h after pouring, weighed, wrapped in aluminium foil, and stored in a thermal container until the beginning of the immersion.

## 2.2. Immersion solutions

The aggressive solution is a mix of organic acids found in liquid manure: acetic, propionic and butyric. The concentrations of the acids in the solution were the ones found by Kunz et al. (2009) in a previous study of pig manure storage. Table 2 presents the composition of the aggressive solution in mg/L. The solution was renewed at the end of each of the four weeks of the test. Plain water saturated with lime was used as the control solution.

Table 2. Composition of the acid solution.

Acids	Acetic(CH <sub>3</sub> COOH)	Propionic(C <sub>2</sub> H <sub>6</sub> O <sub>2</sub> )	Isobutyric(C <sub>4</sub> H <sub>8</sub> O <sub>2</sub> )
Concentration (mg/L)	2,300	460	46

## 2.3. Experimental procedure

Three mortar samples were tested in each of the experiments conducted. In total, 45 mortars were produced. The immersion experiment started 35 days after demoulding the mortars. They were immersed in the aggressive solution and stored at controlled room kept at 26 °C ± 1 °C for four weeks. The solid-liquid volume ratio was 0.067. At the end of each week, the mortars were removed from the solution, wiped to eliminate superficial water, weighed, and left to dry in an oven at 105°C for 3 days. Then the samples were weighed once again and immersed in a renewed solution. This set of procedures is called a cycle. The compressive strength tests were conducted according to ABNT NBR 5739:2007 right before the immersion, at the end of cycle 2 and after cycle 4, thus providing the compressive strength profile along the test. Weighing the mortars provided the mass loss and the drying provided the mortars water absorption profile. Control mortars were also kept in the control solution at 25 °C during the whole experiment.

## 3. Results and discussion

### 3.1. Flow table test

The values in the flow table test were 337mm for Control, 287mm for NS and 245mm for NS+SF. It was clear that the use of NS and NS+SF caused lower values of consistency when compared to the control mortar, most probably due to their great fineness. Nevertheless, it was possible to note a better cohesion in the mortars with additions (NS and NS+SF), what could indicate better-compacted hardened structure. This result agrees with that obtained by Quercia et al. (2012) in a study with amorphous nano-silica.

### 3.2. Mass loss

Table 3 presents the accumulated mass loss along the cycles. The mortars with NS+SF showed the lowest mass loss values along the 4 cycles.

Table 3. Accumulated mass loss.

Sample	Cycle 1	Cycle 2	Cycle 3	Cycle 4
Control	0,76%	3,05%	5,65%	9,58%
NS	0,75%	3,07%	5,63%	8,74%
NS+SF	0,51%	1,9%	4,06%	7,76%

The results confirm what can be seen in Fig. 1 (a, b and c), which shows the aspect of each type of mortar after the first cycle, after the fourth cycle, and the samples immersed in control solution. It could be observed that the control mortar was more deteriorated, presenting more aggregates on the surface and more leaching of the cement paste. Reactions between the cement matrix and organic acids tend to produce soluble to highly soluble in water calcium

and aluminium salts (Bertron et al. (2004)). NS and NS+SF appeared less deteriorated thus confirming the lowest mass loss values observed along the experiment.

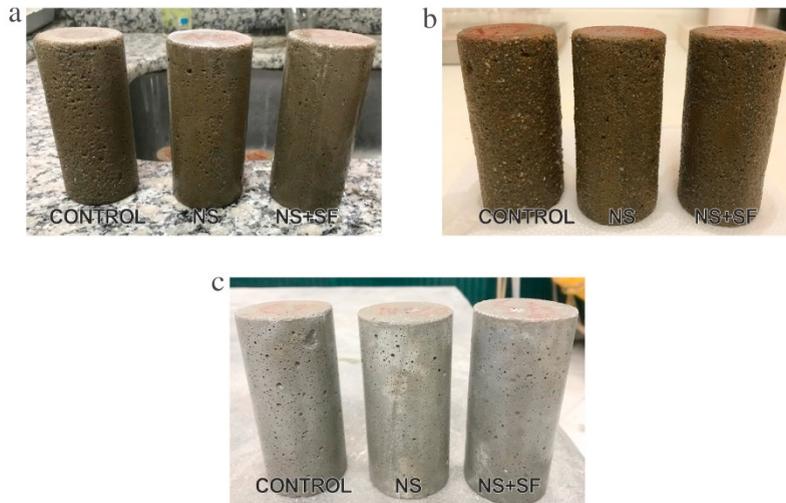


Fig. 1. (a) mortars after Cycle 1; (b) mortars after Cycle 4; (c) mortars in control solution.

### 3.3. Compressive Strength

Table 4 shows the compressive strength mean values and standard deviation (SD) obtained. The samples indicated by (C) refer to the ones immersed in the control solution and the ones indicated by ACID refer to the ones immersed in the acid solution. It can be observed that the results of compressive strength presented low values of SD.

Table 4. Compressive strength results.

		35 days	50 days	65 days			35 days	50 days	65 days
CONTROL (C) (MPa)	Mean (MPa)	24,58	26,43	27,55	CONTROL ACID (MPa)	Mean (MPa)	-	21,46	18,42
	SD	0,96	0,36	0,51		SD	-	0,99	1,38
NS (C) (MPa)	Mean (MPa)	28,15	30,08	31,19	NS ACID (MPa)	Mean (MPa)	-	24,06	23,07
	SD	0,23	0,72	0,42		SD	-	0,56	1,09
NS+SF (C) (MPa)	Mean (MPa)	30,42	34,04	35,72	NS+SF ACID (MPa)	Mean (MPa)	-	30,49	28,23
	SD	0,53	0,67	0,76		SD	-	1,40	0,70

Fig. 2 shows the evolution of the compressive strength along the 65 days, with the results expressed as mean values. An increment in compressive strength could be observed in all the mortars immersed in control solution, probably due to continuous development of cement hydration and formation of C-S-H.

When in control solution, NS+SF developed a compressive strength 29,65% higher than the control mortar at 65 days, whereas NS developed a 13,21% higher value. Therefore, the use of SF and nano-silica improved mechanical strength. The higher development of mechanical strength in mortars with additions may be related to the pozzolanic reactions of silica fume and nano-silica as well as the interactions between them (Nili et al. (2010), Senff et al. (2010b)). It was also observed that the gain in strength was higher within the first two weeks than in the last two weeks, what could be attributed to the lower speed of the cement hydration reactions and the lower amount of  $\text{Ca}(\text{OH})_2$  available for the pozzolanic reactions in the microstructure (Guerrero et al. (2000)).

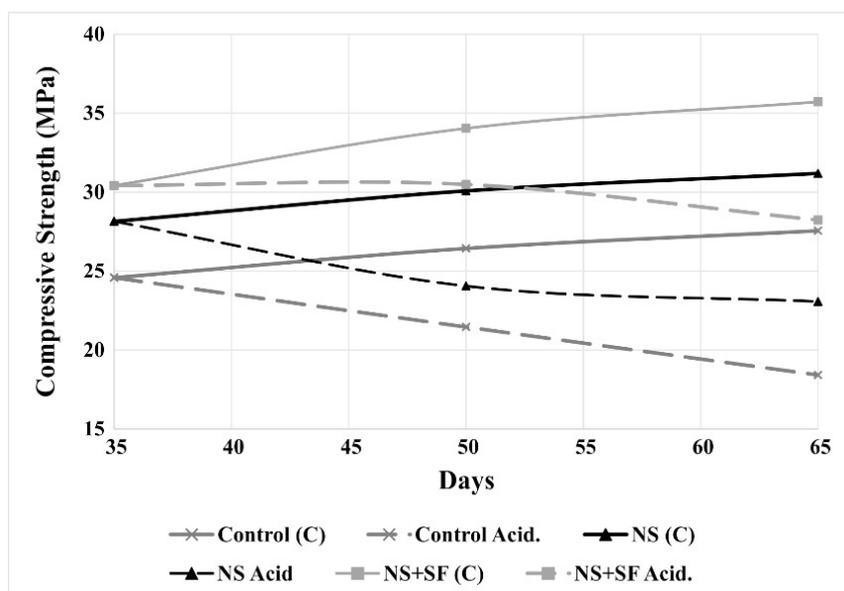


Fig. 2. Compressive strength evolution.

The effect of the acid solution caused a decrease in compressive strength in all types of mortar. This compressive strength profile could be related with the leaching of the cement paste due to the corrosiveness of the pig manure environment (Sánchez et al. (2009)). A decrease of 25,06% was observed between the first and the last test performed in the control mortar, a decrease of 18,04% in NS and a decrease of 7,19% in NS+SF. Thus, NS+SF presented the lowest loss of strength, indicating the best performance to acid solution. This result agrees with that obtained by Hendi et al. (2017). They also concluded that the simultaneous use of silica fume and nano-silica is more effective than their use separately. The use of SF in conjunction with NS may cause pore refinement, better-compacted material and an improved C-S-H (Mendes et al. (2017)).

Massana et al. (2013) conducted an experiment with different types of cement and fly ash, exposing mortars to pig manure for 60 months. The results showed a gain in strength for 24 months and then a decrease in compressive strength after 36 months. A similar result occurred with the NS+SF mortar, which when in acid solution, retained strength in the first two cycles, and eventually showed a loss of strength in the end of experiment. Still they presented the lowest loss of strength.

It was also possible to relate the mass loss results with those of compressive strength, since a greater mass loss could be noted when compressive strength decreased.

### 3.4. Water Absorption

The water absorption measured in all the samples corroborated the results observed in the compressive strength and mass loss tests (Fig. 3). The values of water absorption observed in NS and NS+SF in the control solution were respectively 5,12% and 21,73% lower than those observed in the control mortar. When in acid solution, the water absorption in mortars with mineral additions were more similar to the control mortar, 1,52% lower in NS and 11,74% lower in NS+SF. Hence, the effect of the mineral additions in water absorption when in acid solution was not as remarkable as when in control solution. Nevertheless, the best performance was observed in NS+SF, probably due to the filler effect caused by the interaction between the SF and NS.

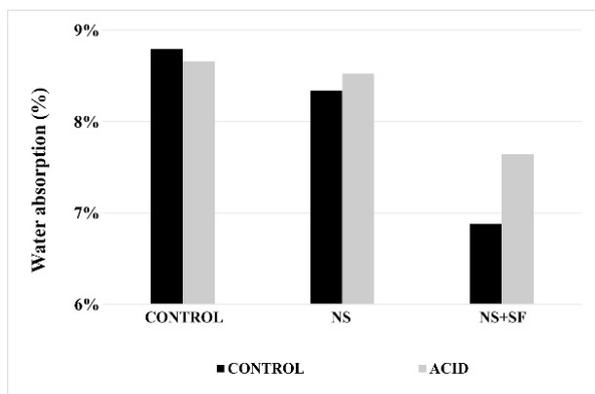


Fig. 3. Water absorption values.

#### 4. Conclusions

The durability of cement mortars containing silica fume and nano-silica exposed to organic acids present in pig manure were investigated in the present study. Three types of mortars were made and submerged in a solution with high concentrations of organic acids. Compressive strength, mass loss and water absorption were measured along the experiment.

The flow table test showed that the additions tend to decrease the mortars consistency. Better cohesion could also be observed, what could indicate better compacting of the hardened material. Mortars with NS and SF presented the lowest water absorption, even when in acid solution. The difference between water absorption values of mortars with both NS and SF in control solution and in acid solution is the highest. This could indicate that the acid solution increased significantly the samples total porosity in this type of mortar, probably due to acid attack and leaching of  $\text{Ca}(\text{OH})_2$ . The use of NS in conjunction with SF showed to be more positive than the use of nano-silica alone, providing the best results concerning compressive strength, mass loss and water absorption. Cement mortars with the use of nano-silica and silica fume even showed a thinner layer of deterioration after the immersion, indicating a better resistance to organic acids.

Therefore, it appears that silica fume and nano-silica in cement mortars as an addition is beneficial to cement mortars, concerning pore refinement, reduction of leaching, reduction of mass loss and increase of compressive strength, hence better resistance to exposure to the organic acids present in pig manure. As a result, the use of these mineral additions could enhance the lifetime of cementitious materials used in pig houses.

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