



Nutritional and quality changes in piglet concentrate affected by the mix

[Mudanças nutricionais e de qualidade no concentrado de leitão afetadas pela mistura]

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ABSTRACT

The aim of this study was to evaluate the reasons of quality deviation of a concentrate from a predefined standard. Five treatments were established: T1 - Control, standard concentrate formulation (SCF); T2 - PXMore5, SCF with more 5% vitamin-mineral premix (VMP); T3 - PXLess5, SCF with less 5% VMP. All three treatments used a 400kg batches in an INTECNIAL mixer; T4 - FeedMixer, SCF using a 4,000kg batch in an IMOTO mixer; T5 - PremixMixer, SCF using a 1,200kg batch in an MUYANG mixer. For each treatment, bags of 20 kg were stored in three storage places for four months. Water activity of concentrate was affected by temperature and air relative humidity in different storage places. Regarding the kind of mixer, the greatest variation in concentration of crude protein, mineral residue, copper, zinc, and selenium was observed in the PremixMixer. Adjustments are imperative in the handling and use procedures of this kind of mixer to meet the quality requirements required in the concentrate production. Analyzing the effect of the mineral-vitamin premix level, no difference could be defined with the evaluated parameters.

Keywords: homogeneity, mixers, swine feed

RESUMO

O objetivo deste estudo foi avaliar as razões do desvio de qualidade de um concentrado de um padrão predefinido. Foram estabelecidos cinco tratamentos: T1 - controle, concentrado com formulação padrão (CFP); T2 - PXMais5, CFP com 5% a mais de vitaminas e minerais da pré-mistura (PVM); T3 - PXMenos5, CFP com 5% a menos de PVM. Todos esses três tratamentos utilizaram lotes de 400kg em um misturador INTECNIAL; T4 - FeedMixer, CFP usando um lote de 4.000kg em um misturador IMOTO; T5 - PremixMixer, CFP usando um lote de 1.200kg em um misturador MUYANG. Para cada tratamento, sacos de 20kg foram armazenados em três ambientes distintos por quatro meses. A atividade de água do concentrado foi afetada pela temperatura e umidade relativa do ar em diferentes locais de armazenamento. Em relação ao tipo de misturador, a maior variação na concentração de proteína bruta, resíduo mineral, cobre, zinco e selênio foi devido ao PremixMixer. Ajustes são imperativos nos procedimentos de manuseio e uso desse tipo de misturador para atender aos requisitos de qualidade exigidos na produção de concentrado. Ao se analisar o efeito do nível da pré-mistura de vitaminas e minerais, nenhuma diferença pôde ser definida com os parâmetros avaliados.

Palavras-chave: alimentação para suínos, homogeneidade, misturadores

INTRODUCTION

For the supply of a complete feed for the animals, mixing the ingredients is a primary operation in the rations production, and necessary when the

ingredients are combined (McCoy *et al.*, 1994). Its objective is to obtain a uniform distribution of the components through a flow generated ordinarily by mechanical processes (Bühler and Degussa, 2006). The more similar the density,

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granulometry, and ease of flow of the ingredients, the better the quality and stability of the mixture (Bühler and Degussa, 2006).

The homogeneity of the ration has significant importance in animal production systems because it provides for all animals the consumption of the same proportions of the nutrients that compose the feed, thus satisfying their nutritional requirements (Teixeira *et al.*, 2012).

The industries accept as a technical parameter for dispersion of ingredients a coefficient of variation (CV) equal to or less than 10% (Johnston and Southern, 2000), however, the Ministry of Agriculture, Livestock and Supply (MAPA), through the Normative Instruction no. 65 (Brasil, 2006), establishes that the mixer homogenization efficiency must have a coefficient of variation not exceeding 5%. Values greater than 5% should be investigated and corrected. The homogenization efficiency assessment can be carried out using indirect indicators, such as *microtracers*, micro minerals, and others.

In the concentrates production, the mixing time is considered the main factor that affects its efficiency, however, other factors such as the mixer model, use and wear and tear of the equipment's mixing components, cleaning, number of turns in a mixing cycle, speed of helicoids, lift blades or threads, the particle size of ingredients, sampling, and choice of an indicator can also influence, therefore, a specific mixing time is not advisable for all equipments (Ciftci and Ercan, 2003; Clark *et al.*, 2007).

Pig diet formulation require several nutritional requirements to be met. Formulating compositions with different compounds, becomes a challenge, since they can interact with each other and with the environment during its shelf life. Also, the ingredients used are influenced by both raw material and storage periods as well as relative humidity and temperature to which they are subjected.

The aim of this study was to evaluate different mixing systems in the quality deviation of a concentrate from a pre-defined standard.

MATERIAL AND METHODS

This study was performed between the beginning of the summer solstice and one month after the autumn equinox of the southern hemisphere (from December 26, 2017, to April 26, 2018) and is an approach to evaluate the quality of concentrates for piglets. The treatments were established in a feed industry located in the Western region of Santa Catarina state (in the south of Brazil) that annually produces 200 thousand tons of products for feeding pigs and poultry such as rations, concentrates, premixes, and supplements. The industry followed good production practices according to Normative Instruction (NI) 04/2007 (Brasil, 2007), updated by Normative Guideline 03/2020 (Brasil, 2020), following NI 65/2006 (Brasil, 2006). The product evaluated was a concentrate recommended for piglets in the pre-initial phase used in the proportion of 400 kg per ton of ration, with a shelf life of 90 days, and it was sent to commercial warehouses serving pig farmers located in the south and central-west regions of Brazil over distances up to 1,200km.

Five treatments were established to evaluate the effect of the quality variation of the concentrates when stored (under monitored conditions) during 120 days in three different places (cities) and variations in the concentrates production. The treatments were:

T1 - Control: Standard concentrate formulation (Table 1), using a 400kg batch in an INTECNIAL mixer (model MH-400, Brazil);

T2 - PXMore5: Standard concentrate formulation (Table 1) with more 5% mineral-vitamin premix for piglets in the supplement (Table 2), using a 400kg batch in an INTECNIAL mixer (model MH-400, Brazil);

T3 - PXLess5: Standard concentrate formulation (Table 1) with less 5% mineral-vitamin premix for piglets in the supplement (Table 2), using a 400kg batch in an INTECNIAL mixer (model MH-400, Brazil);

T4 - FeedMixer: Standard concentrate formulation (Table 1), using a 4.000 kg batch in an IMOTO mixer (model MH-5000, Brazil);

T5 - PremixMixer: Standard concentrate formulation (Table 1), using a 1.200 kg batch in an MUYANG mixer (model SJHS8, China).

Table 1. Ingredients and chemical composition of concentrate

Concentrate ingredients	g/kg		
Whey partially demineralized, spray dried	450.00		
Soybean, micronized processed at 90°C	168.75		
Soybean meal, ground	137.50		
Supplement (as shown in Table 2)	112.50		
Yeast, spray dried	81.25		
Blood plasma, spray dried	50.00		
Chemical composition, kg	T1, T4 and T5	T2	T3
Moisture, g	50.86	50.85	50.87
Metabolizable Energy, Kcal	3400	3400	3400
Crude Protein, g	299.64	299.55	299.74
Dairy Protein, g	54.35	54.35	54.35
Lactose, g	324.00	324.00	324.00
Ether Extract, g	44.72	44.72	44.71
Crude Fiber, g	13.01	13.05	12.97
Mineral Residue, g	106.08	106.15	106.01
Calcium, g	20.00	20.00	20.00
Available Phosphorus, g	11.75	11.75	11.75
Total/digestible Lysine, g	27.95/26.75	27.95/26.75	27.96/26.75
Total/digestible Methionine, g	9.33/9.21	9.33/9.22	9.33/9.21
Total/digestible Threonine, g	17.0/16.4	17.0/16.4	17.00/16.4
Total/digestible Tryptophan, g	4.58/4.05	4.58/4.05	4.58/4.05
Total/digestible Valine, g	12.74/11.62	12.73/11.62	12.74/11.62
Total/digestible Met+Cys, g	13.68/13.60	13.68/13.60	13.68/13.60
Sodium, g	6.25	6.25	6.25
Copper, mg	50.00	52.50	47.50
Zinc, mg	6250.00	6250.00	6250.00
Chromium, µg	499.99	499.99	499.99
Organic Selenium, mg	0.375	0.394	0.356
Vitamin A, IU	37500.0	39375.0	35625.0
Biotin, µg	250.00	262.50	237.50
Choline, mg	875.00	875.00	875.00
Density, g/l	578.520	578.398	578.642

The mineral-vitamin supplement added to the treatments (Table 2) was prepared using the MUYANG horizontal mixer in batches of 1.800kg.

The supplements and concentrates were produced on the same day. The warranty levels declared by the concentrate producer and required following the MAPA standard (Normative Instruction 38/2015 of 10/27/2015) for product registration was described in Table 3.

The helicoid mixers used in this study have only one axis, however, that axis has two metal straps that intersect inside the mixer, making the raw material inside it makes a back-and-forth movement, that is, besides turning, the product goes to the center and returns to the walls to make

the homogenization. The helicoid mixer allows 50% to 100% filling degree, while the paddle mixer has as main advantages the time to produce a batch (faster), besides being able to work with only 30% of the volume capacity of the mixer (filling degree 30 to 100%).

The technical specifications of the mixing systems adopted to produce the supplements and concentrates are shown in Table 4. In order to previously characterize the potential of the different mixing systems used to provide a uniform product, the monitoring in evaluations performed between the years 2015 to 2017 using manganese as a marker was systematized. The mixing efficiency is presented through the coefficients of variation and standard deviations ($CV \pm SD$).

Table 2. Supplement composition (g/kg) used in the preparation of the concentrates

Ingredients (g/kg)	T1, T4 and T5	T2	T3
Mineral-vitamin premix for piglets*	66.6667	70.0000	63.3333
Granulated dicalcium phosphate	181.4444	180.7964	182.0925
Calcitic limestone	152.4444	151.9000	152.9889
Granulated iodized salt	33.7778	33.6571	33.8984
Zinc Oxide 80%	74.0722	73.8077	74.3368
Ground soybean meal	73.6558	73.3924	73.9184
L-Lysine HCl 99%	123.5333	123.0921	123.9745
DL- Methionine 99%	56.2444	56.0436	56.4453
L-Threonine 98,50%	46.0444	45.8800	46.2089
L-Tryptophan 99%	10.8222	10.7836	10.8609
Benzoic acid (E210)	66.6667	66.4286	66.9048
Anti-caking agent (E551**)	26.6667	26.5714	26.7619
Antioxidant (E310, E321, E330)	13.3333	13.2857	13.3810
Flavoring agent (Coconut + Vanilla)	12.2222	12.1786	12.2659
Sweeteners (E952, E954, E959)	6.6667	6.6429	6.6905
Zinc amino acid complex 16%	5.5556	5.5357	5.5754
Iron amino acid complex 22%	3.0278	3.0170	3.0386
Selenium yeast complex	22.2222	22.1429	22.3016
Organic chromium 3.4%	4.4444	4.4286	4.4603
Choline chloride 60%	14.9333	14.8800	14.9867
Protease enzyme / Phytase enzyme	4.4444 / 0.4444	4.4286 / 0.4429	4.4603 / 0.4460
Beta-glucanase and xylanase enzyme	1.1111	1.1071	1.1151

*Vitamin A/D₃, vitamin A, vitamin E, menadione sodium bisulfite, vitamin B₁, vitamin B₂, vitamin B₆, vitamin B₁₂ - 1%, calcium pantothenate, biotin, niacin, folic acid - 80%, copper sulphate pentahydrate, ferrous sulphate, manganese sulfate, calcium iodate monohydrate, zinc sulfate monohydrate, sodium selenite. **Particle size above 100 nm GMD.

Table 3. Concentrate warranty levels (per kg) according to the package label

Parameter	Level	Value	Parameter	Level	Value
Moisture, g	Max	100.00	Lysine, g	Min	20.00
Crude Protein, g	Min	216.00	Methionine, mg	Min	5400.00
Ether Extract, g	Min	35.00	Vitamin A, IU	Min	30000.00
Crude Fiber, g	Max	15.00	Vitamin D ₃ , IU	Min	4000.00
Mineral Residue, g	Max	130.00	Vitamin E, IU	Min	200.00
Calcium, g	Max	28.00	Vitamin K ₃ , mg	Min	7.50
Calcium, g	Min	14.00	Vitamin B ₁ , mg	Min	8.00
Phosphorus, g	Min	7000.00	Vitamin B ₂ , mg	Min	20.00
Copper, mg	Min	40.00	Vitamin B ₆ , mg	Min	15.00
Iron, mg	Min	310.00	Vitamin B ₁₂ , mcg	Min	70.00
Manganese, mg	Min	120.00	Pantothenic Acid, mg	Min	40.00
Iodine, mg	Min	3.00	Niacin, mg	Min	70.00
Zinc, mg	Min	5000.00	Folic Acid, mg	Min	6.00
Selenium, mg	Min	0.50	Biotin, mg	Min	0.20
Chromium, mg	Min	0.40	Choline, mg	Min	700.00

Table 4. Technical specification of the three horizontal mixers used in concentrates (INTECNIAL, IMOTO and MUYANG) and supplements (MUYANG) production and uniformity tests

Technical specification	INTECNIAL Model MH-400	IMOTO Model MH-5000	MUYANG Model SJHS8
Treatment	T1, T2 and T3	T4	T5
Mixer structure using single central rotor coupled with	double shaft ribbon type blender	double shaft ribbon type blender	six transverse axes of double shaft paddle type blender
Central rotor rotation, rpm	27.0	35.5	34.0
Total capacity, kg	400	5000	2000 (or eight m ³)
Load used, kg	400	4000	1200 or 1800
Mixture end point, seconds	300	120	90
Maximal admitted CV, %	5.0	5.0	5.0
CV ± SD, %	3.24±0.94	4.91±1.45	4.40±1.04
Number of evaluations	5	17	12
Evaluated load, by weight %	from 30 to 100	from 20 to 80	from 20 to 90
Mixture end point, seconds	300 to 360	120	90 to 120

The bagging of the concentrates for each treatment was performed in valved bags with properties to ensure the filling, conservation, and protection requirements according to Normative Instruction 22/2009 (Brasil, 2009). The technical characteristics of the bags used were: a) flexible continuous welding packaging, b) making with polyethylene films (low density, linear low density, and high density, respectively, LDPE, PELBD, and HDPE), arranged in multilayers using the lamination technology, c) dimensions of 504x710x378 mm (width, height and fan-folded width), d) laminar thickness of 110µm, e) fan-folded depth on the left and right side 64mm, f) external friction coefficient of 0.32 (COF according to ASTM D1894) and, g) white (external) and black (internal) pigment coating.

After the production and packaging of the concentrates, the bags were coded to identify the treatments and transported proportionally in quantity produced and under standardized conditions to three marketing warehouses: Chapecó/SC, Araquari/SC, and São Gabriel do Oeste/MS (SGO).

At each destination, the bags were stored on pallets in an airy environment, protected from light and heat sources. The storage conditions for 120 days in the three places were monitored using a thermohygrometer (AKSO, AK28 model, São Paulo, Brazil) used to record, three times a day, the temperature and air relative humidity (ARH).

Fortnightly, during the 120 days of storage of the concentrates, 300g samples were collected from

bags of each treatment at the storage sites for chemical analysis of moisture, crude protein, ether extract, crude fiber, mineral residue, in addition to calcium, and phosphorus (Table 5). The sampling procedures were performed according to the recommendations of the Brazilian Compendium of Animal Feed (Compêndio..., 2009) compatible with The Codex General Guidelines on Sampling CAC/GL 50-2004 (Codex..., 2004). The analyzes were performed in a laboratory belonging to the industrial complex of the company responsible for the quality control of raw materials and elaborated products. The ingredients used in the concentrates were previously analyzed according to the quality control routine established by the ration industry.

Also, 300g samples were collected on the day of production and every fifteen days of storage for analysis of minerals by Atomic Absorption Spectrophotometry (AAS) by Inductively Coupled Argon Plasma (ICP) as recommended methodology no. 38 of the CBAA (Compêndio..., 2009), corresponding to method 985.01 AOAC (Official..., 2006) ICP/OES (Inductively Coupled Plasma/Optical Emission Spectroscopy). In an outsourced laboratory certified by National Institute of Metrology, Quality and Technology (INMETRO) for ABNT NBR ISO/IEC 17025:2017 (Associação..., 2017) the minerals Copper (Cu), Iron (Fe), Manganese (Mn), Zinc (Zn), Selenium (Se), Chromium (Cr), Sodium (Na), Magnesium (Mg) and Potassium (K) were analyzed. Before making the concentrates, the ingredients used were previously sampled for minerals analysis according to the quality control routine established by the feed industry.

Table 5. Analyzes performed on concentrates and their definitions by the methods of Brazilian Compendium of Animal Feed (CBAA), the analytical deviation admitted, analysis amplitude, and the AOAC equivalence

Parameter	CBAA method	Analytical deviation admitted (%)*	Analysis amplitude *	AOAC International Status
Moisture and volatiles, g/kg	53/2013	291/x + 3	41 – 141	934.01 (1995)
Crude protein, g/kg	46/2013	167/x + 1	62 – 849	990.03 (1995)
Ether extract, g/kg	14/2013	154/x + 4	9 – 226	920.39 (2006)
Mineral material, g/kg	05/2013	101/x + 2	11 – 991	942.05 (2007)
Crude fiber, g/kg	19/2009	206/x + 8	18 – 250	978.10 (2006)
Calcium, g/kg	04/2013	33/x + 6	3 – 392	935.13 (2007)
Phosphorus, g/kg	23/2013	23/x + 4	2 – 233	965.17 (2007)
Mineral analysis by Inductively Coupled Plasma / Optical Emission Spectroscopy (ICP/OES): AOAC 985.01 (2006)				
Sodium, g/kg	38/2009	13	4 – 150	956.01 (2006)
Magnesium, mg/kg	38/2009	35	1641 – 23200	984.27 (2007)
Iron, mg/kg	38/2009	17	908 – 8177	984.27 (2007)
Zinc, mg/kg	38/2009	8	580 – 9810	984.27 (2007)
Potassium, mg/kg	38/2009	31	580 – 7800	956.01 (2006)
Manganese, mg/kg	38/2009	13	294 – 5535	917.04 (2006)
Copper, mg/kg	38/2009	10	173 – 5527	947.03 (2006)
Selenium	38/2009	20	-	996.16 (2006)
Chromium	38/2009	-	-	990.08 (2006)

* Based on 16,500 interlaboratory tests in conventional laboratory analyzes.

To perform the analyzes, the percentages of the absolute difference between the calculated values and the observed values of the evaluated nutrients were first calculated. The percentages of absolute difference for day 0 on the shelf were presented in a table. Subsequently, the averages of the percentages of the absolute difference were calculated (disregarding the value of day 0 on the shelf), by treatment and storage place, with the storage place considered as repetition. These means were subjected to analysis of variance for the model containing the effects of location and treatment. The details of the treatment effect were performed using the Tukey test for multiple comparison of means, whenever the F test detected a significant effect ($P \leq 0,05$). The analyzes were performed using the GLM procedure of the SAS (SAS, 2012).

The model evaluated in the variance analysis was:
 $Y_{ij} = \mu + L_i + T_j + e_{ij}$,

Meaning: Y_{ij} is the mean of the percentage of the absolute difference between the calculated value and the observed value of the i-th storage place with the j-th treatment; μ is the fixed effect of the overall mean; L_i is the effect of the i-th storage place; T_j is the effect of the j-th treatment; e_{ij} is the experimental error, supposed to be random, normal, homoscedastic and independently distributed. To evaluate the effect of treatments on

the percentage of samples within the warranty levels, Fischer's Exact Test was applied.

RESULTS

The highest temperature average was observed in SGO (27.19 ± 0.2949), while the highest relative humidity average was in Araquari (80.88 ± 0.937) with the lowest temperature average (22.15 ± 0.2017). In Araquari, the highest temperature and relative humidity averages were observed in January (24.87 ± 0.2264 and 80.88 ± 0.937 , respectively). In Chapecó the highest temperature average was observed in January (26.65 ± 0.2593) and the highest relative humidity average was observed in March (61.29 ± 1.1451). While in SGO the highest temperature and relative humidity averages occurred in March (27.19 ± 0.2949 and 78.41 ± 0.12944 , respectively).

Tables 6 (moisture, crude protein, ether extract, and crude fiber), 7 (macro minerals), and 8 (micro minerals) express the average values that represent the percentage difference between the value analyzed for each treatment and the value calculated for different parameters. These percentages of difference between the values may be due to the production process of the concentrates, sampling process, and laboratory analysis or these two factors.

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In all evaluated parameters, it was not possible to establish a significant difference ($P>0.05$) between the Control treatment and the treatments with the addition or removal of 5% of mineral-vitamin premix (PXMore5 and PXLess5). This phenomenon is also especially valid for micro

minerals (Table 8). Assuming a perfect, standard, and uniform sampling in addition to the use of analysis techniques under standardized conditions, the calculated variability represents, theoretically, the effects of the treatments.

Table 6. Mean and standard error of the percentage of absolute difference between the observed and the calculated values for chemical composition

Treatment	Variable, %			
	Moisture	Crude Protein	Ether Extract	Crude Fiber
Control	5.922±0.759	12.83±0.18 ^B	5.771±0.633	9.322±0.292
PXMore5	5.852±0.650	13.33±0.47 ^B	5.930±0.507	8.717±0.637
PXLess5	5.515±0.390	11.93±0.28 ^B	5.703±0.408	10.48±0.54
FeedMixer	3.311±0.608	13.34±0.45 ^B	3.748±0.516	9.746±1.024
PremixMixer	5.321±1.273	24.21±0.24 ^A	5.377±0.678	12.76±1.57
Mean	5.184±0.395	15.13±1.23	5.306±0.300	10.20±0.51
Pr>F	0.1251	<0.0001	0.0621	0.0884

^{A, B} Means followed by different letters in the column differ by Tukey's test ($P<0.05$); Pr>F: probability.

Table 7. Mean and standard error of the percentage of absolute difference between the observed and the calculated values for mineral residue and macro minerals

Treatment	Variable, %			
	Mineral Residue	Calcium	Phosphorus	Sodium
Control	3.735±0.373 ^B	17.17±0.53 ^{AB}	8.114±1.264	11.73±1.33
PXMore5	4.463±0.096 ^B	20.33±0.38 ^A	9.737±0.832	16.53±1.87
PXLess5	4.306±0.258 ^B	17.81±2.08 ^A	6.674±0.482	11.11±0.94
FeedMixer	4.328±0.799 ^B	12.69±0.51 ^B	9.442±1.090	13.78±2.23
PremixMixer	16.45±1.04 ^A	7.250±0.794 ^C	8.261±0.497	18.67±1.89
Mean	6.656±1.331	15.05±1.30	8.446±0.445	14.36±1.00
Pr>F	<0.0001	<0.0001	0.1442	0.0647

^{A, B} Means followed by different letters in the column differ by Tukey's test ($P<0.05$); Pr>F: probability.

For the crude protein, it was found that the PremixMixer treatment mixer had greater variability ($P<0.05$) (Table 7), in the same way for mineral residue (Table 8) and especially for copper, zinc, and selenium (Table 8). For mineral residue, the PremixMixer treatment mixer showed greater variations in concentrations ($P<0.05$), but an opposite effect was found for the variability of calcium ($P<0.05$), while other macro minerals evaluated showed no significant difference ($P>0.05$). The variation in the percentage of the difference between the observed and the calculated values for crude fiber, mineral residue, and micro minerals has a high positive correlation (values always above 0.900) to each other, that is, when the value of the deviation of one parameter increases the other parameter also increases. Positive correlations were observed between the deviations of copper and zinc (0.9928), copper

and selenium (0.9357), and zinc and selenium (0.9542). However, the opposite occurs for chromium, that is, the greater the variability of the determined value of a parameter, the less variability in chromium deviations is observed. The correlations observed were between selenium and chromium (-0.9197), copper and chromium (-0.8922), and zinc and chromium (-0.9192). Thus, according to the data analyzed, chromium presents a technological challenge since, for maximum uniformity in parameters, chromium presents high variability in its concentration. There is a need to establish some operational procedures to reduce these occurrences. The high correlation between the micro mineral distribution deviations between them indicates that if the supplement is evenly distributed when the mixture is made, all the minerals contained in it are evenly distributed.

Table 8. Means and standard errors of the percentage of absolute difference between the observed and the calculated values for micro minerals.

Treatment	Variable, %			
	Copper	Zinc	Selenium	Chromium
Control	13.25±5.09 ^B	26.09±4.57 ^B	112.74±18.3 ^{AB}	30.69±10.3
PXMore5	13.10±4.97 ^B	17.43±3.10 ^{BC}	77.57±20.6 ^B	30.92±5.93
PXLess5	11.52±1.43 ^B	21.73±4.28 ^B	73.88±21.2 ^B	36.44±4.27
FeedMixer	4.784±1.32 ^B	6.906±2.78 ^C	76.30±9.31 ^B	39.78±2.59
PremixMixer	195.18±5.89 ^A	119.27±3.69 ^A	175.56±19.5 ^A	18.97±1.65
Mean	47.57±19.8	38.29±11.0	103.21±12.5	31.36±2.90
Pr>F	<0.0001	<0.0001	0.0036	0.2307

^{A,B,C} Means followed by different letters in the column differ by Tukey's test (P<0.05); Pr>F: probability.

The percentages of analysis results that reached the value established on the label according to the treatment applied and the absolute frequency in agreement with the specifications are shown in Table 9. It is observed that, for most parameters, the percentage of samples that are within the warranty range was high, showing a good mix quality. The most critical parameters were for chromium and selenium, and only in the concentration of chromium there was a difference between treatments (P<0.05), with the PremixMixer differing from the others. The average value for reaching the specifications was 41.4% for chromium and 65.8% for selenium.

When linking the results of the percentage of the relative contribution calculated of the ingredients on parameters based on the quality control before mixing the concentrates with those in table 9, it can be seen that 95.2% of the concentration of the ether extract is homogeneously distributed in the soybean meal and micronized soybean indicating that they are uniformly mixed in the concentrates. Similarly, the supplement is evenly distributed, contributing to 78.5% of the mineral residue concentration and 46.6% of the calcium concentration.

Table 9. Percentage of analysis results that reached the value established on the label depending on the treatment applied and the absolute frequency in agreement with the specifications

Analysis	Sig	Control	PXMore5	PXLess5	Feed Mixer	Premix Mixer	Freq
Moisture	NS	100.0	100.0	100.0	100.0	100.0	123/123
Crude Prot.*	NS	100.0	100.0	100.0	100.0	95.5	119/120
Ether Extr.	NS	100.0	100.0	100.0	100.0	100.0	124/124
Crude Fiber*	NS	92.0	92.0	84.0	88.0	100.0	113/124
Mineral Res.*	NS	100.0	100.0	100.0	100.0	88.0	122/125
Calcium*	NS	96.0	100.0	92.0	96.0	100.0	121/125
Total Phosphorus	NS	100.0	100.0	100.0	100.0	100.0	125/125
Copper	NS	100.0	100.0	100.0	100.0	100.0	46/46
Zinc	NS	100.0	100.0	100.0	100.0	100.0	46/46
Chromium**	S	33.3 ^b	25.0 ^b	30.0 ^b	30.0 ^b	88.9 ^a	19/46
Selenium*	NS	75.0	42.9	66.7	55.6	88.9	28/42

Sig: significance; Freq: indicates the relation between the number of analyzes comply with the specification and the total number of analyzes performed. * It indicates that the specifications were not 100% satisfied. **^{a,b} Percentages followed by different letters in the row differ by Fischer's Exact Test (P≤0.05). NS indicates not significant in Fisher's Exact Test (P>0.05).

DISCUSSION

The results obtained with the different mixers determined that there is potentially an effect on the mixture quality, with a different performance for the PremixMixer system. For this system, the

difference between the calculated value and the value obtained in the laboratory analyzes for crude protein, mineral residue, and some micro minerals was greater when compared to the other treatments. Associated with this observation, the smallest variation between the calculated and the

analyzed values for calcium indicated that there was a deficiency in the homogeneous distribution of the supplement and powdered whey, which corresponds to 45% in the mixture in the concentrate. Considering all parameters evaluated simultaneously, the smallest difference between the calculated and the analyzed allows the use of lower safety coefficients for the concentration of nutrients in the concentrate's formulation. This can mean cost reduction, less environmental impact due to the reduction in the excretion of excess nutrients by the piglets, while there is an assurance that nutritional requirements are being met, even when these nutrients are needed in very low concentrations. However, among the essential micro minerals, for the concentration of selenium the largest deviation was detected in relation to the calculated one, regardless of the evaluated mixing system, although this deviation was more accentuated in the PremixMixer and Control mixing systems. Chromium concentration did not reach the level specified on the label and the difference between calculated and analyzed values was also dependent on the mixing system.

An adjusted standard operating procedure was applied to each mixing system. The three mixers used in the industry to produce the concentrates have constant homogenization efficiency tests, mainly using manganese (Mn) as a marker. The results of these uniformity tests, previously performed, were within the requirements of the MAPA normative instructions with a coefficient of variation (CV) of less than 5% when the specific concentrate was produced.

Uniform mixing is essential to produce products for animal feed in order to achieve technical parameters in accordance with established marketing standards (nutritional levels, stability, and safety of its constituents included in the recommended proportion and shelf life). Due to the increased use of low inclusion ingredients, efficiency in the mixing process becomes even more important. The inclusion of enzymes in very low concentration, such as phytase, the need for greater uniformity in the mixture of diets and concentrates increases, in order to obtain an adequate nutritional balance for calcium and phosphorus in animal metabolism (Johnston and Southern, 2000). Piglets in the nursery phase showed worse performance only when they received rations with CV in the mixture above 28.4% (Traylor et al., 1994). However, Groesbeck

et al. (2007) presented data evaluating weight gain and feed conversion of weaned piglets, indicating that a coefficient of variation (CV) of 12% is the maximum limit of variation in the mixing uniformity of the rations provided. Herman and Behnke (1994) expressed the results of mixing tests in four categories: excellent, good, reasonable, and poor according to the obtained CVs. Mixtures with a CV lesser than 10% were classified as excellent, mixtures with a CV of 10 to 15% were considered good, CV of 15 to 20% was considered reasonable, and mixtures with a CV greater than 20% were classified as poor. Approximately 5% of the variations seen in a feed uniformity test occur due to sampling and laboratory analysis (Martin, 2005). In this study, with the removal or addition of 5% of mineral-vitamin premix to the concentrate, it was not possible to establish a difference when compared to the control treatment, and this phenomenon was also especially valid for micro minerals. Assuming a standard, perfect, and uniform sampling in addition to the use of analysis techniques under standardized conditions, the calculated variability theoretically represents the effects of the applied treatments. According to Rocha *et al.* (2015) as Mn, Cu, Zn and Cl are intrinsic in the feed ingredients, so the use of Manganese Sulphate (MnSO₄), Copper Chloride (CuCl₂), Zinc Sulphate (ZnSO₄), and Sodium Chloride (NaCl) may cause errors in the results interpretation, and some of these errors may be caused by the granulometry of the indicator used (Wilcox and Unruh, 1986).

The different mixers used in the present study indicate that there is potentially an effect on the quality of the mixture, as observed with the results of the analysis of the minerals, however, considering the standard operating procedure used, these effects are minimized.

Each kind of mixer has a different ideal mixing time. For example, horizontal mixers generally tend to have a shorter ideal mixing time than vertical mixers. With the wear and tear of the mixer tapes and drills, mixing times need to be increased to meet proper mixing standards. Diets containing low-density ingredients may also require extra time to achieve the desired mixing uniformity (McCoy, 1994).

The selection of ingredients with similar particle sizes should improve the efficiency and result of

mixing and a more uniform mixture of the final product (Clark *et al.*, 2007; Groesbeck *et al.*, 2007). Thus, minimizing particle sizes is not only important for optimizing growth performance of pigs but also for enhancing feed manufacturing processes (Amornthewaphat *et al.*, 1998). Each mixer analysis will be unique due to the formulation of the diet, the particle size of the raw ingredients, wear and tear on the mixer parts, mixer cleanliness, individual sampling, mixing time, and the marker chosen for mixer uniformity (Clark *et al.*, 2007).

Despite the different environmental conditions identified in the four months of the experimental period and the evolution of Aw values, there was no consequence or practical effect on the visual alteration of the mixtures and there was no demand for recall. Thus, under controlled conditions of product handling, aiming at detailed monitoring in different environments, no effects were observed that would determine the possibility of the occurrence of recall.

To ensure the product quality, priority must be given to monitoring the production routine, performing frequent monitoring of relevant quality parameters, including the mixture quality.

CONCLUSION

When analyzing the effect of the mixer kind, it was found that the PremixMixer system showed greater variation in the concentration of crude protein, mineral residue, copper, zinc, and selenium. Adjustments in the handling and use procedures of this mixing system are necessary to satisfy the required quality in the concentrate production. Based on the evaluation procedures applied, it was not possible to obtain a change in the concentration of the premix when varying in the amplitude of 5% more or less in the supplement.

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REFERENCES

- AMORNTHAWAPHAT, N.; BEHNKE, K.C.; HANCOCK, J.D. *Effects of particle size and mixing time on uniformity and segregation in pig diets*. Manhattan, Kansas: Kansas State University, 1998. p.261-263.
- ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS. *NBRISO/IEC 17025. Requisitos gerais para a competência de laboratórios de ensaio e calibração*. 3.ed. São Paulo: ABNT, 2017. 32p.
- BRASIL. Ministério da Agricultura, Pecuária e Abastecimento - MAPA. Instrução Normativa Nº 65: regulamento técnico sobre os procedimentos para a fabricação e o emprego de rações, suplementos, premixes, núcleos ou concentrados com medicamentos para os animais de produção. Brasília, 21 de novembro de 2006.
- BRASIL. Ministério da Agricultura, Pecuária e Abastecimento - MAPA. Instrução Normativa Nº 22: regulamenta a embalagem, rotulagem e propaganda dos produtos destinados à alimentação animal. Brasília, 02 de junho de 2009.
- BRASIL. Ministério da Agricultura, Pecuária e Abastecimento - MAPA. Instrução Normativa Nº 38: altera a Instrução Normativa MAPA nº 42, de 16 de dezembro de 2010, que estabelece critérios e procedimentos para a fabricação, fracionamento, importação e comercialização dos produtos isentos de registro. Brasília, 27 de outubro de 2015.
- BRASIL. Ministério da Agricultura, Pecuária e Abastecimento - MAPA. Orientação Normativa Nº 03: modelos de formulários utilizados na fiscalização de estabelecimentos de produtos destinados à alimentação animal, bem como os manuais para seus preenchimentos. Brasília, 16 de junho de 2020.
- BRASIL. Ministério da Agricultura, Pecuária e Abastecimento – MAPA. Instrução Normativa Nº 04: regulamento técnico sobre as condições higiênico-sanitárias e de boas práticas de fabricação para estabelecimentos fabricantes de produtos destinados à alimentação animal e o roteiro de inspeção. Brasília, 23 de fevereiro de 2007.

- BÜHLER and DEGUSSA. Qualidade de mistura. 2006. Disponível em: <https://www.buhlergroup.com/content/buhlergroup/global/pt/homepage.html>. Acessado em: 7 jul. 2018.
- CIFTCI, Y.; ERCAN, A. Effects of diets of different mixing homogeneity on performance and carcass traits of broilers. *J. Anim. Feed. Sci.*, v.12, p.163-171, 2003.
- CLARK, P.M.; BEHNKE, K.C.; POOLE, D.R. Effects of marker selection and mix time on the coefficient of variation (mix uniformity) of broiler feed. *J. Appl. Poult. Res.*, v.16, p.464-470, 2007.
- CODEX ALIMENTARIUS. Codex Committee on Methods of Analysis and Sampling. CAC/GL 50-2004. The codex general guidelines on sampling. Roma: FAO/WHO, 2004. 73p.
- COMPÊNDIO brasileiro de alimentação animal - (CBAA). São Paulo: Sindicato Nacional da Indústria de Alimentação Animal, 2009.
- GROESBECK, C.N.; GOODBAND, R.D.; TOCKACH, M.O. *et al.* Diet mixing time affects nursery pig performance. *J. Anim. Sci.*, v.85, p.1793-1798, 2007.
- HERRMAN, T.; BEHNKE, K. *Testing mixer performance*. MF1172. Kansas State University Agricultural Experiment Station and Cooperative Extension Service Bulletin, Manhattan, KS: Kansas State University, 1994. 4p.
- JOHNSTON, S.L.; SOUTHERN, L.L. The effect of varying mix uniformity (simulated) of phytase on growth performance, mineral retention, and bone mineralization in chicks. *Poult. Sci.*, v.79, p.1485-1490, 2000.
- MARTIN, S. Mixing. In: SCHOFIELD, E.K. (Ed.). *Feed manufacturing technology*. Arlington: American Feed Industry Association, Inc., 2005. 670p.
- MCCOY, R.A.; BEHNKE, K.C.; HANCOCK, J.D.; MCELLHINEY, R.R. Effect of mixing uniformity on broiler chick performance. *Poult. Sci.*, v.73, p.443-451, 1994.
- OFFICIAL methods of analysis of AOAC international. 18.ed. Gaithersburg: AOAC, 2006.
- ROCHA, A.G.; MONTANHINI, R.N.; DILKIN, P. *et al.* Comparison of different indicators for the evaluation of feed mixing efficiency. *Anim. Feed. Sci. Technol.*, v.209, p.249-256, 2015.
- SAS INSTITUTE INC. System for Microsoft Windows. Release 9.4. Cary, NC, USA, 2002-2012. (cd-rom).
- TEIXEIRA, M.M.; RIZZO, R.; DETMANN, E. *et al.* Avaliação da qualidade da mistura de ração em misturador horizontal considerando a homogeneidade dos ingredientes. *Encicl. Biosfera.*, v.8, p.123, 2012.
- TRAYLOR, S.L.; HANCOCK, J.D.; BEHNKE, K.C. *et al.* Mix time affects diet uniformity and growth performance of nursery and finishing pigs. *KSU Swine Day Rep.*, p.171-175, 1994.
- WILCOX, R.; UNRUH, D. *Feed manufacturing problems-feed mixing times and feed mixers*. Manhattan, KS: Kansas State University, 1986. (Bulletin MF-829, Kansas State University Agricultural Experiment Station and Cooperative Extension Service).