Animal Feed Science and Technology 169 (2011) 79–85

Contents lists available at ScienceDirect

Animal Feed Science and Technology

journal homepage: www.elsevier.com/locate/anifeedsci

Fiber analysis: Evaluation of screen printing fabric filters bags by three statistical approaches

M. de J. Marichal*, A.I. Trujillo, M. Cadenazzi, G. Arias

Departamento de Producción Animal y Pasturas. Facultad de Agronomía. Universidad de la República Oriental del Uruguay. Garzón 780. 12900 – Montevideo, Uruguay

ARTICLE INFO

Article history:
Received 26 August 2010
Received in revised form 26 May 2011
Accepted 10 June 2011

Keywords:
Fiber analysis
Filter bags
Bland–Altman method
Deming method

ABSTRACT

Measurements of fiber fractions using the Ankom filter bag system, and four types of filter bags were compared with results obtained employing the procedure indicated by the Association of Official Analytical Chemists (AOAC). To assess agreement between procedures, three statistical approaches were used. Twenty-three materials differing widely in cell wall content, and composition were evaluated. Filter bags evaluated were Ankom F_{57} (porosity: 25 μm), and three types of bags made in our laboratory with polyester screen printing fabric, porosities 45 μm (B_{120}), 36 μm (B_{140}), and 23 μm (B_{150}). Amylase neutral detergent fiber organic matter basis (aNDFom), acid detergent fiber organic matter basis (ADFom), and sulfuric acid lignin (Lignin (sa)) values obtained using crucibles (C) were compared with data obtained using F_{57}, B_{120}, B_{140} or B_{150} bags, and fiber values obtained with F_{57} were compared with data obtained employing B_{120}, B_{140} or B_{150} bags. Statistical approaches used were analysis of variance (ANOVA), regression analysis (Deming method), and the Bland–Altman method of differences. In the ANOVA, no differences ($P>0.18$) were observed, between measurements of aNDFom, ADFom, and Lignin (sa) performed employing the AOAC, or the Ankom procedure using any type of bag. In the regression analysis, in all associations tested, the 0.95 confidence interval of the slope of the regressions contained the value 1, and Pearson correlations ($P<0.001$) were greater than 0.94. In aNDFom, ADFom, and Lignin (sa) comparisons, five, three, and two of the seven regressions did not include 0 in the 0.95 confidence interval of de intercept. In the Bland–Altman method, proportion of differences between limits of agreement, ranged from 0.91 to 1.0, 0.91 to 0.96, and 0.74 to 0.96, respectively, in aNDFom, ADFom and Lignin (sa) method comparisons. The statistical approaches evaluated, suggested a reasonable degree of agreement between aNDFom, ADFom, and Lignin (sa) measurements performed by the AOAC, and the Ankom Fiber Analyzer procedure, when F_{57} or B_{120} bags were used. This would suggest that using these procedures in sequential analysis, it is unlikely to yield erroneous values of the analytes. The Bland–Altman method resulted in a sensitive method, to identify differences among analytical procedures. Results suggest that, in the description of fiber protocols using the Ankom Fiber Analyzer, it is crucial to specifically describe filter bags employed.

Abbreviations: AOAC, Association of Official Analytical Chemists; aNDFom, amylase neutral detergent fiber organic matter basis; ADFom, acid detergent fiber organic matter basis; Lignin (sa), sulfuric acid lignin; F_{57}, Ankom filter bags; B_{120}, filter bags constructed from screen printing fabric: porosity 45 μm; B_{140}, filter bags constructed from screen printing fabric: porosity: 36 μm; B_{150}, filter bags constructed from screen printing fabric porosity: 23 μm; C, crucibles; ANOVA, analysis of variance.

* Corresponding author. Tel.: +598 23572344; fax: +598 23543460.
E-mail addresses: mariadejesus.marichal@gmail.com, marichal@fagro.edu.uy (M.d.J. Marichal), anatrujister@gmail.com (A.I. Trujillo), gabrielaarias@hotmail.com (G. Arias).

0377-8401/$ – see front matter © 2011 Published by Elsevier B.V.
doi:10.1016/j.anifeedsci.2011.06.005

1. Introduction

The determination of fiber using the detergent system (Van Soest et al., 1991) is a routine analysis in most laboratories involved in feedstuffs analysis, and animal nutrition research. The procedure indicated by the Association of Official Analytical Chemists (AOAC, 2007) to determine neutral detergent fiber, acid detergent fiber, and sulfuric acid lignin, involves a number of extraction steps in a conventional refluxing apparatus, followed by the collection of residues in fritted glass crucibles. To enhance laboratory operations, and reduce labor costs a semi-automated procedure of analysis, was developed (Ankom Technology Corp., Fairport, NY), and is widely used. In this procedure solvent extractions are carried out in the same vessel, and samples are encapsulated in filter bags.

To compare fiber results obtained by different procedures, a variety of statistical approaches have been used. Approaches reported are regression analysis (Fay et al., 2005; Senger et al., 2008), mean tests for paired samples (Fay et al., 2005), analysis of variance using different experimental designs (Berchielli et al., 2001; Cassida et al., 2007; Ferreira and Mertens, 2007), and the Bland–Altman method of differences (Bland and Altman, 1986). This method is a simple parametric approach based on graphical techniques, and simple calculations.

The Ankom Fiber Analyzer employs filter bags (F_{57}) that are produced exclusively in the United States. This may impose a constraint to utilize the Ankom procedure in countries outside the United States, because local custom regulations, and taxes may increase considerably the cost of F_{57} bags.

To overcome this restriction, research has been performed to evaluate alternative filter bags. Berchielli et al. (2001) measured neutral, and acid detergent fiber of five materials (sugar cane, Brachiaria grass, corn silage, citrus pulp, and cattle feces) employing the AOAC (2007), and the Ankom procedure. Four types of filter bags were evaluated, the F_{57} bag (porosity: 25 micron), and bags made with Ankom *in situ*, or a similar polyester fabric (same size as F_{57}, porosity: 50 μm). Differences ($P<0.05$) in neutral, and acid detergent fiber were detected when results obtained with filter bags were compared with data obtained using crucibles. However, in all feedstuffs, bag type did not affect ($P>0.05$) fiber values. These results suggest that similar values may be obtained using F_{57}, or alternative bags. The preparation of bags with materials easily, and widely available like screen printing fabric, may contribute to facilitate the adoption of the filter bag system.

The objectives of this study were to compare the Ankom filter bag system, using four types of filter bags, with the AOAC procedure, and to assess agreement between procedures using three statistical approaches.

2. Materials and methods

2.1. Feedstuffs

Twenty-three materials, selected to represent a wide range of neutral detergent fiber, acid detergent fiber, and sulfuric acid lignin content, were evaluated. Materials were selected from samples sent for analysis to the Laboratory of Animal Nutrition of the Faculty of Agronomy (University of the Republic, Montevideo, Uruguay). Samples of three roughages, seven pastures (collected in vegetative stage), five preserved forages, six agro industrial byproducts, and two cereal grains were evaluated. All materials were dried in a forced air oven at 60 °C for 48 h, and ground in a Willey mill to pass a 2 mm sieve. Dry matter was determined in all feedstuffs according to AOAC (2007).

2.2. Fiber analysis procedures

Fiber analyses were performed following two procedures, the official AOAC (2007), and the Ankom Technology Corp (Fairport, NY). In both procedures, the neutral detergent solution included a heat-stable alpha amylase, and omitted the sodium sulphite. The neutral detergent and acid detergent fiber analyses were performed sequentially (Van Soest et al., 1991).

In the AOAC procedure (Official Methods 2002.04, and 973.18; 2007), samples were extracted individually in Berzelius beakers on a reflux rack, and filtered through standard coarse fritted disk Gooch crucibles (Pyrex® Gooch crucible, high form, 50 mL, porosity: 40–60 μm). Sequential acid detergent fiber organic matter basis (ADFom) analysis was conducted by rinsing carefully all neutral fiber residues present in the crucible, into a new beaker where acid detergent solution was added. After acid detergent extraction, each beaker was filtered into a crucible for sulfuric acid lignin (Lignin (sa)) determination. At the end of this procedure, crucibles were dried in a 100 °C forced-draft oven, and weighed; then residues in crucibles were ashed at 600 °C to obtain residual ash.

In the Ankom procedure (Ankom Technology Corp., Fairport, NY), extractions were performed using filter bags, and the Ankom^{200} Fiber Analyzer equipment. Sulfuric acid lignin was determined using the Daisy^{II} Incubator (Ankom Technology Corp., Fairport, NY), and the 08/05 Ankom protocol, including the final ash of entire bags. Four types of bags were evaluated, the F_{57} Ankom filter bag (porosity: 25 μm), and three other type of bags constructed in our laboratory (Animal Nutrition Laboratory, Faculty of Agronomy, University of the Republic, Montevideo, Uruguay). Bags were constructed from N-free monofilament polyester screen printing fabrics (Sefar Inc., Switzerland) that differed in pore size: PET 1000 120-34-W (porosity: 45 μm) (B_{120}), PET 1000 140-31-W (porosity: 36 μm) (B_{140}), and PET 1000 150-34-W (porosity: 23 μm) (B_{150}).

All analyses were performed in quadruplicated. In the Ankom procedure analytical replicates were obtained distributing four samples of each feedstuff in eight batches of analysis, because the capacity of Ankom Fiber Analyzer allows incubating

24 bags at a time, and four blanks were included in each run. In both procedures, residual ashes were subtracted from the neutral and acid detergent fiber values, and fractions were expressed in ash-free dry matter basis (aNDFom and ADFom, respectively).

2.3. Data analysis

Homogeneity of variance and normality were tested using, respectively, the HOVTEST of PROC GLM (SAS, 2000; Bartelett's test), and the PROC UNIVARIAT (SAS, 2000; Shapiro–Wilk test, alpha = 0.05). As Lignin (sa) was not normally distributed the arcsine transformation was applied to the data, and normality was checked using the same previously stated procedure.

Results of aNDFom, ADFom, and Lignin (sa) analysis performed with crucibles (C) (AOAC, 2007), were compared with those obtained using the Ankom procedure, and the four types of bags. Besides, data obtained using the F_{57} bag were compared with fiber values obtained with B_{120}, B_{140} or B_{150} bags. Agreement between procedures was evaluated by analysis of variance (ANOVA), regression analysis, and the Bland–Altman method.

The ANOVA was performed in a completely randomized design, using the PROC GLM procedure (SAS, 2000). Treatments were AOAC procedure using crucibles (C), and the Ankom procedure using bags B_{120} (B_{120}), B_{140} (B_{140}), or B_{150} (B_{150}). Means were considered to differ (Tukey test) when $P<0.05$.

In the regression analysis, the Deming procedure was employed, using the Jacknife method to estimate the standard errors (Linnet, 1993). The slope, intercept, standard errors, and 0.95 confidence intervals were calculated using the MedCalc software (version 11.5.1). Pearson correlations were performed using the PROC CORR of SAS (SAS, 2000). Correlations were considered significant at $P<0.05$.

In the Bland–Altman method (Altman and Bland, 1983) means of the paired measurements of fiber (aNDFom, ADFom, or Lignin (sa)) obtained by each combination of filtering devices evaluated (*x*-axis), were plotted against their difference (*y*-axis). The average of differences between filtering devices, and the 95% limits of agreement of the mean of differences (2 standard deviations; more precisely 1.96) were calculated, and superimposed on the plot. The number of points that fell within the limits of agreement were then observed, and recorded.

3. Results

The aNDFom, ADFom, and Lignin (sa) content of feedstuffs, obtained with the procedures evaluated in this study, are presented in Tables 1–3, respectively. As expected, feedstuffs presented a wide range of cell wall content, and composition. The aNDFom, ADFom, and Lignin (sa) content of feedstuffs evaluated, ranged from 94 to 827, 16 to 607, and 0 to 235 g kg^{-1} DM, respectively.

Table 1
Amylase neutral detergent fiber organic matter basis (g kg^{-1} DM) obtained using crucibles, and four types of filter bags.

	C[1]	F_{57}	B_{120}	B_{140}	B_{150}
Agro industrial byproducts					
Citrus pulp	265.1	267.3	253.1	238.3	240.4
Rice bran	275.6	294.5	190.9	193.4	185.8
Corn defatted germ	372.3	394.7	362.0	363.9	371.0
Sunflower expeller	472.0	484.0	463.3	454.6	444.8
Malt sprouts	704.6	652.0	652.0	644.0	624.0
Dried brewers' grains	829.3	726.3	710.0	703.2	721.9
Pastures					
Orchard grass	671.7	611.3	605.5	604.8	593.2
Fescue	638.2	582.5	601.9	591.4	599.6
Ryegrass	579.4	513.0	528.1	523.4	526.8
Lotus Maku	475.9	511.7	411.8	418.1	418.2
White clover	312.5	388.0	270.4	282.7	275.3
Chicory	336.2	370.9	326.0	326.9	328.4
Plantain	344.3	358.2	322.7	334.8	327.4
Rouhages					
Barley straw	827.7	774.1	808.4	797.2	814.4
Rice hulls	762.4	745.1	769.0	739.0	807.5
Soybean hulls	685.1	635.6	646.3	643.0	642.0
Preserved forages					
Alfalfa hay	434.6	490.3	351.5	377.8	400.1
Foxtail millet haylage	735.7	646.6	647.0	673.9	670.5
Sweet sorghum silage	498.3	467.9	464.9	464.1	466.6
Corn silage	467.5	433.3	446.3	445.9	447.2
Sorghum silage	667.7	648.6	646.8	633.8	638.7
Cereal grains					
High moisture corn	277.5	169.0	111.0	123.6	111.3
Sorghum	195.5	123.2	93.8	106.1	109.7

[1] C: Gooch crucible; F_{57}: Ankom filter bag; B_{120}, B_{140}, B_{150}: bags made with screen printing fabric (porosities: 45, 36, and 23 μm, respectively).

Table 2
Acid detergent fiber organic matter basis (g kg^{-1} DM) obtained using crucibles, and four types of filter bags.

	C[1]	F_{57}	B_{120}	B_{140}	B_{150}
Agro industrial byproducts					
Citrus pulp, dried	166.1	185.5	176.7	171.2	161.5
Rice bran	109.7	76.7	75.9	76.3	66.2
Corn defatted germ	93.8	85.6	79.4	82.9	75.9
Sunflower expeller	298.0	327.7	297.4	303.5	303.4
Malt sprouts	183.3	177.5	187.4	182.8	178.7
Dried brewers' grains	259.5	228.6	200.7	205.1	212.3
Pastures					
Orchard grass	326.2	314.7	309.8	301.9	298.1
Fescue	304.7	291.2	306.2	294.5	296.7
Ryegrass	288.4	284.0	283.3	279.9	271.6
Lotus Maku	309.5	317.0	308.6	304.0	290.9
White clover	169.6	199.3	175.8	178.4	172.7
Chicory	195.1	268.3	240.9	241.3	243.3
Plantain	195.1	268.3	240.9	241.3	243.3
Rouhages					
Barley straw	490.1	465.1	486.1	483.6	478.4
Rice hulls	532.4	557.0	550.4	537.3	609.2
Soybean hulls	405.3	434.3	436.7	438.5	445.0
Preserved forages					
Alfalfa hay	287.4	282.5	268.1	267.7	270.8
Foxtail millet haylage	369.6	360.0	357.6	375.0	467.0
Sweet sorghum silage	255.1	293.7	278.8	265.8	268.0
Corn silage	264.1	282.1	269.2	268.6	261.5
Sorghum silage	349.0	349.0	342.5	339.3	340.0
Cereal grains					
High moisture corn	62.7	23.5	26.0	25.1	15.5
Sorghum	65.3	66.4	51.4	54.8	53.4

[1] C: Gooch crucible; F_{57}: Ankom filter bag; B_{120}, B_{140}, B_{150}: bags made with screen printing fabric (porosities: 45, 36, and 23 μm, respectively).

Table 3
Sulfuric acid lignin (g kg^{-1} DM) obtained using crucibles and four types of filter bags.

	C[1]	F_{57}	B_{120}	B_{140}	B_{150}
Agro industrial byproducts					
Citrus pulp, dried	43.1	50.3	51.0	23.9	28.2
Rice bran	38.1	43.0	27.5	27.2	17.6
Corn defatted germ	14.8	12.1	8.7	9.1	0.0
Sunflower expeller	109.4	103.0	92.2	96.3	84.7
Malt sprouts	36.2	18.9	16.6	17.7	5.3
Dried brewers' grains	160.7	151.9	142.8	145.5	134.9
Pastures					
Orchard grass	46.5	84.5	38.4	33.7	26.8
Fescue	35.6	60.3	31.1	23.0	22.2
Ryegrass	33.8	25.1	17.3	14.8	6.1
Lotus Maku	184.6	166.8	168.5	152.6	217.8
White clover	73.5	52.3	38.9	39.7	30.7
Chicory	143.4	167.5	120.1	113.1	129.8
Plantain	78.8	125.1	95.7	94.8	84.3
Roughages					
Barley straw	80.4	67.1	64.7	69.2	62.0
Rice hulls	169.0	68.2	48.9	55.7	44.1
Soybean hulls	40.2	47.4	33.5	27.1	21.6
Preserved forages					
Alfalfa hay	75.0	76.7	48.1	65.7	55.1
Foxtail millet haylage	68.0	58.4	54.8	48.7	44.8
Sweet sorghum silage	55.8	50.5	48.2	38.3	31.8
Corn silage	35.4	38.1	27.3	32.8	19.8
Sorghum silage	38.5	31.5	27.6	22.3	17.3
Cereal grains					
High moisture corn	34.6	2.1	4.7	4.4	0.0
Sorghum	23.0	22.1	21.1	23.0	14.0

[1] C: Gooch crucible; F_{57}: Ankom filter bag; B_{120}, B_{140}, B_{150}: bags made with screen printing fabric (porosities: 45, 36, and 23 μm, respectively).

Table 4
Results of the analysis of variance of amylase neutral detergent fiber organic matter basis (aNFDom), acid detergent fiber organic matter basis (ADFom), and acid sulfuric lignin (Lignin (sa)) obtained using crucibles, and four types of filter bags.

	C[1]	F_{57}	B_{120}	B_{140}	B_{150}	SEM	P=
aNDFom	515.2	490.4	468.0	464.5	464.5	198.0	0.88
ADFom	260.0	266.0	258.3	257.0	261.0	132.1	0.99
Lignin (sa)	65.2	59.5	47.3	44.9	36.5	9.5	0.18

[1] C: Gooch crucible; F_{57}: Ankom filter bag; B_{120}, B_{140}, B_{150}: bags made with screen printing fabric (porosities: 45, 36, and 23 μm, respectively).

Table 5
Regression analysis (Deming method) relating amylase neutral detergent fiber organic matter basis (aNDFom), acid detergent fiber organic matter basis (ADFom), and sulfuric acid lignin (Lignin (sa)) values obtained with crucibles, and four types of filter bags.

		b_0[b]	SE	0.95 CI[c]	b_1	SE	0.95 CI	R[d]
aNDFom								
C[a]	F_{57}	−2.1	5.0	−12.4–8.2	1.09	0.09	0.91–1.27	0.97
C	B_{120}	7.2	3.1	0.8–13.6	0.95	0.06	0.83–1.08	0.98
C	B_{140}	5.8	3.0	−0.4–12.1	0.98	0.06	0.87–1.10	0.98
C	B_{150}	7.0	3.1	0.46–13.5	0.95	0.06	0.82–1.08	0.98
F_{57}	B_{120}	8.5	2.1	4.0–13.1	0.87	0.04	0.79–0.95	0.98
F_{57}	B_{140}	7.2	2.1	2.8–11.7	0.90	0.04	0.82–0.97	0.98
F_{57}	B_{150}	8.4	2.3	3.5–13.3	0.87	0.04	0.78–0.95	0.98
ADFom								
C	F_{57}	0.5	1.4	−2.5–3.4	0.96	0.05	0.86–1.05	0.97
C	B_{120}	1.6	0.9	−0.2–3.6	0.94	0.03	0.88–0.99	0.98
C	B_{140}	1.5	1.0	−0.5–3.5	0.95	0.03	0.89–1.02	0.98
C	B_{150}	3.6	1.1	1.2–5.9	0.86	0.05	0.75–0.96	0.97
F_{57}	B_{120}	1.2	0.6	0.01–2.5	0.98	0.02	0.93–1.02	0.99
F_{57}	B_{140}	1.0	0.6	−0.2–2.3	0.99	0.03	0.93–1.05	0.99
F_{57}	B_{150}	3.3	0.9	1.2–5.3	0.89	0.05	0.79–0.98	0.98
Lignin (sa)								
C	F_{57}	0.7	0.04	−0.06–0.1	0.93	0.16	0.60–1.26	0.94
C	B_{120}	1.3	0.02	−0.02–0.08	1.00	0.12	0.75–1.25	0.95
C	B_{140}	1.9	0.02	−0.003–0.09	1.00	0.12	0.75–1.24	0.96
C	B_{150}	11.4	0.02	0.07–0.14	0.77	0.11	0.55–0.99	0.95
F_{57}	B_{120}	0.1	0.02	−0.03–0.05	1.08	0.09	0.88–1.26	0.98
F_{57}	B_{140}	0.3	0.02	−0.02–0.06	1.07	0.09	0.88–1.23	0.98
F_{57}	B_{150}	7.3	0.02	0.05–0.12	0.83	0.10	0.62–1.03	0.98

[a] C, Gooch crucible; F_{57}, Ankom filter bag; B_{120}, B_{140}, B_{150}, bags made with screen printing fabric (porosities: 45, 36, and 23 μm, respectively).
[b] b_0, b_1: intercept and slope of regressions
[c] CI: Confidence interval
[d] R: Pearson correlation, P<0.001

In the ANOVA, no differences (P>0.18) were observed, between measurements of aNDFom, ADFom, and Lignin (sa) performed employing the AOAC, or the Ankom procedure using any type of bag (Table 4).

In Table 5, results of the regression analysis (Deming method) are presented. In aNDFom, ADFom, and Lignin (sa) comparisons, five, three, and two out of seven regressions did not include 0 in the 0.95 confidence interval of de intercept. In all associations tested, the 0.95 confidence interval of the slope of the regressions contained the value 1, and Pearson correlations were greater (P<0.001) than 0.94.

In the Bland–Altman analysis, no significant (P>0.18) relationships were detected (Spearman r range: 0–0.7) between means of the paired measurements of fiber (aNDFom, ADFom, or Lignin (sa)) obtained by the each combination of filtering devices, and their difference. Therefore, the assumption of independence was not contradicted by the data in any of the comparisons evaluated. Twenty-one plots (not shown) were analyzed. Averages differences, 95% limits of agreement between each pair of filtering devices, and proportion of values included between limits of agreement are presented in Table 6. In the evaluation of aNDFom, ADFom and Lignin (sa), proportion of differences between limits of agreement ranged from 0.91 to 1.0, 0.91 to 0.96, and 0.74 to 0.96, respectively.

4. Discussion

Ranges of aNDFom, ADFom, and Lignin (sa) content of feedstuffs were in reasonable agreement with values reported by NRC (2000, 2001), Sanderson et al. (2003), and Di Marco et al. (2009), except the aNDFom of dried brewers' grains and malt sprouts, which were greater than quoted by these references. The high content of crude protein in the aNDFom fraction (220 and 180 g kg^{-1} DM, in dried brewers' grains, and malt sprouts, respectively) may explain differences (NRC, 2001). Removing this crude protein from aNDFom resulted in values within the range reported by NRC (2001).

Table 6
Bland–Altman comparison of amylase neutral detergent fiber organic matter basis (aNDFom), acid detergent fiber organic matter basis (ADFom), and sulfuric acid lignin (Lignin (sa) values obtained with crucibles, and four types of filter bags.

	Average of differences[b]	Limits of agreement		Proportion of differences between limits of agreement
		+1.96 SD[c]	−1.96 SD	
aNDFom (g kg^{-1} DM)				
C[a] *vs* F_{57}	23.5	123.6	−76.5	1.00
C *vs* B_{120}	49.8	132.3	−32.6	0.96
C *vs* B_{140}	49.8	121.4	−21.8	0.91
C *vs* B_{150}	46.3	130.3	−37.7	0.91
F_{57} *vs* B_{120}	26.3	119.8	−67.1	0.96
F_{57} *vs* B_{140}	26.3	106.6	−54.0	0.96
F_{57} *vs* B_{150}	22.8	119.3	−58.9	1.00
ADFom (g kg^{-1} DM)				
C *vs* F_{57}	−5.9	49.2	−61.1	0.96
C *vs* B_{120}	1.8	48.6	−45.1	0.96
C *vs* B_{140}	3	48.6	−42.6	0.91
C *vs* B_{150}	−1	70.2	−72.2	0.91
F_{57} *vs* B_{120}	7.7	33.4	−18.1	0.96
F_{57} *vs* B_{140}	8.9	34.1	−16.3	0.96
F_{57} *vs* B_{150}	4.9	108.7	−56.9	0.91
Lignin (sa) (g kg^{-1} DM)				
C *vs* F_{57}	4.1	59.2	−50.9	0.96
C *vs* B_{120}	17	66.4	−32.4	0.91
C *vs* B_{140}	19.1	64.8	−26.6	0.74
C *vs* B_{150}	22.6	74.8	−29.6	0.74
F_{57} *vs* B_{120}	12.8	41.0	−15.3	0.91
F_{57} *vs* B_{140}	19.9	45.3	−15.3	0.91
F_{57} *vs* B_{150}	18.4	109.8	−55.6	0.91

[a] C: Gooch crucible; F_{57}: Ankom filter bag; B_{120}, B_{140}, B_{150}: bags made with screen printing fabric (porosities: 45, 36, and 23 μm, respectively).
[b] Average of differences of each combination of filtering devices evaluated.
[c] SD: Standard Deviation.

The ANOVA analysis, suggested procedures, and filtering devices evaluated, could be interchangeable, and would result in similar aNDFom, ADFom, and Lignin (sa) values.

In the regression analysis, in all the comparisons evaluated and fiber fractions tested, the value of 1 was included in confidence interval of the slopes, and high ($P<0.01$) Pearson correlations were observed. That suggested a strong association between the AOAC, and Ankom (using any of the bags) procedures. In those associations where 0 was not included in the confidence interval of the intercept, methods involved in the comparison could differ, at least by a constant amount. This suggests that calibrations could be produced, to interconvert values of measurements obtained with the B_{150} bag to C values.

In the Bland–Altman approach, neither an over, nor an underestimation of results obtained with one filtering device respect to other could be established, because differences between means, were not all positives or negatives. This approach seemed to be very sensitive to identified differences between procedures. The overall analysis suggested that a reasonable degree agreement among procedures may be achieved, when crucibles, F_{57} or B_{120} bags are used in fiber determination, particularly in aNDFom, and ADFom measurements. Meanwhile, a reasonable lack of agreement was observed when crucibles, B_{140} or B_{150} bags were employed, particularly in Lignin (sa) determination. The lowest agreement between procedures was observed in the measurement of this fraction. This may be related to the small lignin content in the evaluated, and the magnitude of the errors related to the analytical method.

In this study, results agreed with these reported by Komarek (1993), Fay et al. (2005), and Komarek et al. (1993). These authors registered, respectively, similar neutral detergent fiber values in alfalfa, and corn silage using crucibles or F_{57} bags, and similar ADFom-C and ADFom-F_{57} in eight forages, and 19 feedstuffs.

5. Conclusions

The statistical approaches evaluated, suggested a reasonable degree of agreement between aNDFom, ADFom, and Lignin (sa) measurements performed by the AOAC, and the Ankom Fiber Analyzer procedure, when using F_{57} or B_{120} bags. This would suggest that using those procedures, it is unlikely to yield erroneous aNDFom, ADFom, and Lignin (sa) values in sequential analysis. The Bland–Altman method resulted in a sensitive method, to identify differences among analytical procedures. Results implicates that it is crucial that authors, and Laboratories using the Ankom filter bag procedure, accurately state in the description of fiber protocols the filter bags employed.

References

Altman, D.G., Bland, J.M., 1983. Measurement in medicine: the analysis of method comparison studies. Statistician 32, 307–317.

AOAC, 2007. Official Methods of Analysis, 18th ed. Association of Official Analytical Chemists, Washington, D.C.

Berchielli, T.T., de Oliveira Sader, A.P., Tonani, F.L., de Paziani, S., de Andrade, P., 2001. Use of the Ankom system to determine neutral detergent fiber and acid detergent fiber with different filter bags, and sample amounts. Rev. Bras. Zootec. 30, 1572–1578.

Bland, J.M., Altman, D.G., 1986. Statistical methods for assessing agreement between two methods of clinical measurement. Lancet 328, 307–310.

Cassida, K.A., Turner, K.E., Foster, J.G., Hesterman, O.B., 2007. Comparison of detergent fiber analysis methods for forages high in pectin. Anim. Feed Sci. Technol. 135, 283–295.

Di Marco, O.N., Ressia, A.M., Aello, M.S., Arzadún, M., 2009. Digestibility of forage silages from grain, sweet and bmr sorghum types: Comparison of in vivo, in situ and in vitro data. Anim. Feed Sci. Technol. 153, 161–168.

Fay, J.P., Guaita, M., Danelón, J.L., Chifflet, S., Wawrzkiewicz, M., Hernández, H.M., Ross, D., 2005. Evaluation of two procedures to determine acid and neutral detergent fibers in ruminant feeds of the temperate region of Argentina. J. AOAC 88, 995–997.

Ferreira, G., Mertens, D.R., 2007. Measuring detergent fibre and insoluble protein in corn silage using crucibles or filter bags. Anim. Feed Sci. Technol. 133, 335–340.

Komarek, A.R., 1993. A filter bag procedure for improved efficiency of fiber analysis. J. Dairy Sci. 76 (1), 250.

Komarek, A.R., Robertson, J.B., Van Soest, P.J., 1993. A comparison of methods for determining ADF using the filter bag technique versus conventional filtration. J. Dairy Sci. 77 (Suppl. 1), 114.

Linnet, K., 1993. Evaluation of regression procedures for methods comparison studies. Clin. Chem. 39, 424–432.

NRC, 2000. Nutrient Requirements of Beef Cattle updated, 7th ed. National Academy Press, Washington, DC.

NRC, 2001. Nutrient Requirements of Dairy Cattle, 7th ed. National Academy Press, Washington D.C., USA.

Sanderson, M.A., Labreveux, M., Hall, M., Elwinger, G.F., 2003. Nutritive value of chicory, and english Plantain Forage. Crop Sci. 43, 1197–1804.

SAS Institute, 2000. Version release 8.2. SAS Institute Inc., Cary, NC.,C.D.

Senger, C.D., Kozloski, G.V., Bonecarrere, L.M., Mesquita, F.R., Alves, T.P., Castagnino, D.S., 2008. Evaluation of autoclave procedures for fibre analysis in forage and concentrate feedstuffs. Anim. Feed Sci. Technol. 146, 169–174.

Van Soest, P.J., Robertson, J.B., Lewis, B.A., 1991. Methods for dietary fiber, neutral detergent fiber, and nonstarch polysaccharides in relation to animal nutrition. J. Dairy Sci. 74, 3583–3597.