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Lysine reactivity and starch gelatinization in extruded and pelleted canine diets

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Abstract

Fifteen dry adult canine diets (*i.e.*, dinners, extrudates, pellets) were collected from retailers in Wageningen, The Netherlands, and chemically and physically characterized. Quality measurements were lysine *O*-methylisourea (OMIU) reactivity and starch gelatinization degree (SGD). In general, extruded diets had a higher crude fat and starch content than pellets. Mean values for starch gelatinization were higher in pellets and ranged between 0.78 and 0.91. The mean reactive/total lysine ratio in extrudate samples was about 5–10% higher than in pellet samples, suggesting the presence in commercial diets of about 200 g bound lysine/kg in pellets and 120 g/kg in extrudates with bound lysine levels of canine dinners about 170 g/kg. Variation of analysed nutrients in pellets was larger than in extrudates. Inclusion of animal or vegetable ingredients, and the process variables during extrusion or pelleting, are the likely causative factors for the variation in lysine reactivity and starch gelatinization.

Keywords: Extrudate; Lysine; Pellet; Pet food; Starch

Abbreviations: ADF, acid detergent fibre; DM, dry matter; OMIU, *O*-Methylisourea; SGD, starch gelatinization degree

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1. Introduction

Commercial pet foods can be categorized into four basic types of dry, semi-moist, moist and snacks. Dry pet foods comprise the largest segment of the total pet foods sold worldwide and 0.95 of pet diets, in practice, are extruded (Spears and Fahey, 2004). The pet food industry predominantly uses extrusion to manufacture dry pet foods because of the ability to pasteurize, increase digestibility/availability, achieve a desired density and form the products in one application (Douglas, 2006). This high temperature short time process does, however, have detrimental effects on nutritional quality (Björck and Asp, 1983; Cheftel, 1986).

Pelleting and extrusion are thermo-mechanical processes that promote chemical changes such as Maillard reactions between the ε-amino group of lysine and the carbonyl group of other compounds (Björck and Asp, 1983; van Barneveld, 1993) and protein cross-linking reactions (Stanley, 1989; Arêas, 1992). Protein quality can be affected by these reactions since the products formed are not always utilized by the animal when digested and absorbed (Hendriks et al., 1999). In addition, carbohydrate quality may be modified by thermo-mechanical treatments through the gelatinization of starch (Lankhorst et al., 2006) or a shift to the development of resistant starch (Dust et al., 2004). Process conditions used during the pelleting or extrusion process determine digestibility/availability to a large extent. Indeed, it has been established that lysine reactivity was affected in dry commercial canine diets (Williams et al., 2006) and experimental extruded diets (Lankhorst et al., 2006), and to be dependent on the conditions used during diet manufacture. Compared to extrusion cooking, pelleting may generate less shear forces on the feed ingredients and operates at much lower product end-temperatures. Extrusion *versus* pelleting leads to a decrease in N digestibility and an increase in ash absorption in dogs fed diets with a high inclusion level of products of animal origin (Stroucken et al., 1996).

This study investigated variation in total and reactive lysine contents, gelatinization degree of starch and physical properties of dry canine foods commercially available in The Netherlands.

2. Materials and methods

2.1. Canine diets and sample preparation

Fifteen dry adult canine diets were obtained from supermarkets in Wageningen, The Netherlands. Diets included four extruded diets, four pelleted diets and seven dinners. The dinners were composed of differently processed ingredients such as extrudates, puffed cereals and flaked grains. All diets were ground (Retsch ZM100 mill, Retsch BV, Ochten, The Netherlands) to pass a 1 mm sieve and stored in air-tight plastic containers at 4 °C prior to analysis.

2.2. Chemical and physical analysis

The composition of the diets was determined by the standard analysis methodology (AOAC, 1990). Dry matter (DM) was analysed by drying samples to a constant weight at

103 °C; the ash content was determined after combustion at 550 °C and N was determined using the Kjeldahl technique with $CuSO_4$ as a catalyst. Crude fat level was determined using the Berntrop treatment (acid hydrolysis) prior to extraction with petroleum ether in a continuous extractor (Soxhlet).

Starch content and starch gelatinization degree (SGD) were enzymatically (*i.e.*, amyloglucosidase) determined as described by Lankhorst et al. (2006). Total lysine content was determined according to the method described by Hendriks et al. (2002) while *O*-methylisourea (OMIU)-reactive lysine content was determined according to Moughan and Rutherfurd (1996). In the latter method, lysine with a free ε-amino group is converted to homoarginine by the use of OMIU and the reactive lysine is calculated from the molar amount of formed homoarginine. All chemical analyses were in duplicate.

Durability and hardness of the extrudates and pellets were measured using the Holmen (simulation of pneumatic transport during 60 s) and an automatic Kahl device, respectively, as described by Thomas and van der Poel (1996). Specific density was calculated as mean ($n = 12$) quotient of kibble weight to kibble volume. In this calculation for specific density, canine dinners were not included.

2.3. Statistical analysis

The correlation coefficients between the various physical characteristics of the products (*i.e.*, the specific density and the reactive/total lysine ratio) were determined using the correlation procedure of SAS 9.1.3 Service Pack 4 (SAS, 2003).

3. Results

Minimum and maximum values for the nutrient composition of the canine diets were (g/kg DM; range): crude fat 50–120; crude protein 221–317; starch 231–495; ADF 50–90; lysine 8.4–14.6; reactive lysine 6.8–13.0. Moisture content ranged from 56 to 109 g/kg diet. The analysis shows that the fat content in the extrudates was higher than that of the pelleted diets and dinners (Table 1). In general, variation of all nutrient contents in pellets was larger. Levels of starch were also higher in the extrudates (Table 2) but the degree of starch gelatinization was higher in the pellets showing a range as a proportion of 0.78–0.91. Both total and reactive lysine content in the extrudates was higher than in the pellets, causing the reactive/total lysine ratio to be highest. The ratio of OMIU reactive lysine, expressed per unit of total lysine – a property related to the effect of thermal-mechanical processing, was lower in the pellets compared to extrudates. In pellets, the average ratio was 0.80 ($n = 4$) while in the extrudates the ratio was 0.87 ($n = 4$).

Extruded samples were 10% less durable, but had a higher value for hardness, *versus* diets which were pelleted. For example, the range within all extrudates for hardness was 11.3–26.8 and for durability, 69.4–99.8.

The correlation coefficients calculated between components and several physical determinations such as kibble thickness, volume and density mostly resulted clearing no correlation (data not shown). However, the specific density of extrudates had a correlation ($r = 0.59$; $P<0.0001$) with the ratio of reactive to total lysine. In contrast, the pellet den-

Table 1
Nutritional and physical quality of commercial dry canine diets

		Canine diet		
		Extrudate	Pellet	Dinner
n		4	4	7
Dry matter	g/kg food	917 ± 11.6	911 ± 5.6	911 ± 18.2
Ash	g/kg DM[a]	75 ± 5.1	77 ± 13.6	78 ± 6.9
Crude fat	g/kg DM	97 ± 24.9	76 ± 30.8	73 ± 16.8
Crude protein	g/kg DM	276 ± 9.9	267 ± 36.8	245 ± 21.2
ADF	g/kg DM	51 ± 2.5	62 ± 18.7	57 ± 6.2
Starch	g/kg DM	388 ± 49.0	362 ± 91.7	418 ± 63.7
SGD	proportion	0.76 ± 0.05	0.86 ± 0.06	0.79 ± 0.09
Total lysine	g/kg DM	14 ± 1.3	12 ± 0.9	11 ± 1.6
Reactive lysine	g/kg DM	12 ± 1.1	9 ± 1.0	9 ± 2.1
Ratio[b]		0.88 ± 0.05	0.80 ± 0.09	0.83 ± 0.10
Physical property				
Durability	%	88 ± 10.7	95 ± 1.2	ND[c]
Hardness	kg	17 ± 6.7	16 ± 3.5	ND

[a] DM, dry matter; ADF, acid detergent fibre; SGD, starch gelatinization degree.
[b] Reactive/total lysine ratio.
[c] Not determined.

Table 2
Density, starch gelatinization degree (SGD) and lysine characteristics of commercial dry canine extrudates ($n = 4$) and pellets ($n = 4$)

Diet	Food form	Density (g/cm^3)	SGD (proportion)	Total lysine (g/kg DM[a])	Reactive lysine (g/kg DM)	Ratio[b]
1	Extrudate	0.47	0.73	14.6	12.5	0.85
2	Extrudate	0.49	0.76	12.9	10.6	0.83
3	Extrudate	0.57	0.83	12.1	11.3	0.93
4	Extrudate	0.64	0.79	14.5	13.0	0.89
5	Pellet	0.97	0.78	13.0	9.4	0.72
6	Pellet	0.99	0.91	11.5	10.8	0.93
7	Pellet	1.01	0.85	10.9	8.4	0.77
8	Pellet	1.15	0.90	11.7	9.0	0.76

[a] Dry matter.
[b] Reactive/total lysine ratio.

sity was not correlated ($r = -0.18$; P=0.23). For all diets ($n = 15$), the correlation between thickness and reactive/total lysine ratio was 0.58 (P<0.0001).

4. Discussion

Except for total starch and fat content, mean contents for other components in extrudates and pellets, respectively, are similar. Quality measurement for starch modification reveals a degree of starch gelatinization between 0.78 and 0.86, indicating current values at higher extrusion temperatures as reported from studies with experimental conditions during extrusion (Lin et al., 1997; Dust et al., 2004; Lankhorst et al., 2006).

Changes in starch gelatinization are considered to be one of the beneficial effects of thermal processing, and high temperature short time extrusion process increases SGD (Harper, 1978; Björck and Asp, 1983; Lin et al., 1997; Murray et al., 2001). Gelatinization increases susceptibility for amylolytic degradation due to loss of crystalline structure (Holm et al., 1988; Björck et al., 2000; Kishida et al., 2001). Holm et al. (1988) reported a correlation of 0.96 between extent of gelatinization and digestion rate in dogs. Extrusion processing usually results in a more complete gelatinization and disintegration of starch granules than pelleting (Asp and Björck, 1989). However, the possibility cannot be excluded that starch components are used which already have been gelatinized prior to mixing for the pelleting process.

The mean contents of bound lysine (*i.e.*, total lysine minus reactive lysine) in extrudates and pellets were 120 and 200 g/kg, respectively (Table 1). The variation for this quality parameter of pellets, however, was larger than extrudates. In the pellets, for example, a minimum of 70 g/kg occurred while the highest value was 280 g bound lysine/kg (Table 2). The variation in canine foods of different brands may reflect conditions used for the preparation of food ingredients, such as maize grain. Moreover, conditions in food preparation, such as extrusion (Lankhorst et al., 2006) or pelleting (Thomas, 1998), almost certainly affect quality measurements such as lysine damage, since heat and shear are involved in both processes. The ratio of reactive to total lysine was considered a quality property directly affected by technological treatments. It is notable that the lowest ratio of reactive to total lysine occurred in three of four pellets, rather than in the extrudates, investigated here. It is well known that shear and die pressure play a major role in physico-chemical properties of the formed product (Lin et al., 1997; Williams et al., 2006).

Extrusion is a high-pressure treatment at a high moisture level, suggesting that food expansion and subsequently relaxation will occur caused by the pressure drop just after the die. Pelleting, however, is a process whereby food is produced at a much lower moisture level, using a longer die hole where no expansion and hardly any relaxation of the product occurs (Thomas, 1998). In practice, extruder die thickness is smaller than a pellet die. With pelleting, increasing die hole length increases pellet residence time in the die, resulting in improved pellet durability although it may affect lysine reactivity. These examples show that, in agglomerating processes, die design and the correlated sectional expansion index of foods during extrusion (Alvarez-Martinez et al., 1988) may be important determinants of nutrient modification. In addition, it has also been shown that extrusion of low reactive lysine foods may increase the reactive lysine level (Lankhorst et al., 2006). Where steam conditioning, shear and cooling are processes involved in either steam pelleting or extrusion of canine diets, extrusion cooking is followed by a drying and coating process prior to cooling, a process that may also affect the reactive lysine content.

5. Conclusions

Commercial diets vary considerably in levels of modified starch and the ratio of total to reactive lysine. The reactive/total lysine ratio of extrudates was 5–10% higher than pellets. Both the interaction between food ingredient properties (*i.e.*, animal *versus* vegetable ingredients) and process variables during pelleting and extrusion, including die expansion phenomena should be further studied to predict their effects on reactivity of lysine and starch gelatinization in the agglomerating and drying processes, as well as their interaction.

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