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The partitioning of water in aggregates of undigested and digested dietary particles

Allan K. Hardacre [a,b,*], Sia-Yen Yap [a], Roger G. Lentle [a,b], Patrick W.M. Janssen [a], John A. Monro [b,c]

[a] Institute of Food, Nutrition and Human Health, Massey University, Private Bag 11222, Palmerston North 4442, New Zealand
[b] Riddet Institute, Massey University, Private Bag 11222, Palmerston North 4442, New Zealand
[c] Plant and Food Research, Private Bag 11600, Palmerston North 4442, New Zealand

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ABSTRACT

The hydration of fibre particles derived from wheat and wood was quantified, before and after *in vitro* digestion, and compared with fibre particles from the colonic digesta of pigs and from human faeces. Total water and the extra- and intra-particulate water components were determined using a combination of centrifugation, drying, gas pycnometry and image analysis. The water of saturation (W_S) of wood particles and AllBran® measured after *in vitro* digestion was up to double that of wheat fibres after *in vitro* digestion, and increased with particle size and loss of soluble material, but was not associated with the chemical composition of the fibres. Fibre that had undergone *in vitro* gastric digestion and that had been recovered from the colon or faeces, sequestered about 3% of the W_s into intra-particulate spaces, the remainder occupying extra-particulate spaces. The authors speculate that large quantities of fibre must be eaten to sequester toxins that locate into the intra-particulate space.

1. Introduction

Whilst much of the solid matter in the human diet is solubilised during digestion, a significant proportion of insoluble, non-nutrient material traverses the digestive tract to be voided as faeces. The presence of these insoluble particles in the lumen of the gut has an adverse effect on the flow and mixing of digesta and on the rate of digestion (Lentle & Janssen, 2008), but is thought to be important for gastrointestinal health (Marlett, McBurney, & Slavin, 2002). Hence, high proportions of insoluble fibrous particles in the diet are associated with a lower incidence of colonic carcinoma (Larsson, Giovannucci, Bergkvist, & Wolk, 2005) and of diverticulitis (Painter & Burkitt, 1971). The reduction in colonic carcinoma has been related to the ability of particulate material to reduce the transit time of digesta, and to sequester unconjugated bile acids and other potential carcinogens (Vahouny, Tombes, Cassidy, Kritchevsky, & Gallo, 1980; Zacherl, Eisner, & Engel, 2011). The reduction in diverticulitis has been related to the ability of the suspended particles to retain water, increase faecal bulk (William & Olmsted, 1936) and hence facilitate flow during peristaltic contraction. The rise in intra-luminal pressure around a plug of viscous digesta with high solid volume content may be ameliorated by the flow of extra-particulate fluid (Lentle, 2009), although a high proportion of closely packed, finely divided particles may prevent this occurring (Wrick et al., 1983).

The solid components of digesta that remain following enzymatic and fermentative digestion generally contain significant amounts of vegetable matter (Ehle, Robertson, & Van Soest, 1982; Holloway, Tasman-Jones, & Lee, 1978). This material comprises an array of cellular remnants of differing sizes and shapes that consist largely of cellulose along with a variable amount of lignin, the proportions of these two components depending on the type and quantity of food consumed. The proportion of such material in dietary items is generally low, being less than 4% on a dry weight basis (dwb) in most cereals and vegetables (Anderson & Bridges, 1988; Rani & Kawatra, 1994), but is higher in some foods e.g. 12% in asparagus and up to 25% in pears (Bunzel, Seiler, & Steinhart, 2005). Over 40% of dietary cellulose may undergo fermentative digestion in the human gut, but digestibility is reduced when the cellulose is associated with lignin (William & Olmsted, 1936).

Water is held in association with solid particles in digesta (Eastwood & Morri, 1992) either as extra-particulate water that occupies the voids between the packed particles, or as intra-particulate water associated with the chemical structures that form the particle surfaces or when it occupies pores within the matrix of the particle. Intra-particulate water is less mobile than

Abbreviations: dwb, dry weight basis; wwb, wet weight basis; W_S, the weight of water in the saturated fibre; WE, the proportion of extra particulate water; WI, the proportion of intra-particulate water.

* Corresponding author at:Institute of Food, Nutrition and Human Health, Massey University, Private Bag 11222, Palmerston North 4442, New Zealand. Tel.: +64 6 3569099x81422.
E-mail address: a.hardacre@massey.ac.nz (A.K. Hardacre).

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extra-particulate water, so any reduction in the water content of digesta as it traverses the length of the colon (Lentle, Janssen, & Hume, 2009), is more likely to result from absorption from the extra-particulate rather than the intra-particulate fraction (Robertson & Eastwood, 1981b).

The relative proportions of extra- and intra-particulate water fractions in compacted particles will vary with their physical and chemical characteristics. The volume of the voids between particles, and the proportion of extra-particulate water contained therein, will vary with the degree of compaction i.e. the volume fraction (Lentle & Janssen, 2008), and thus will depend on the size, shape and elasticity (Lentle, Janssen, & Hume, 2009) of the particles. The intra-particle water content will also depend on the hydrophobicity or hydrophilicity of the surface material and on the permeability of the solid material within the particle.

The distinction between extra- and intra-particulate water is not always clear. The lumen spaces within intact cellular structures comprising the particles may sequester water, thus augmenting the intra-particulate water fraction (Ferguson & Harris, 1997). Conversely, where the walls of component cells are disrupted, water contained within the cell lumen will not be sequestered and will contribute to the extra-particulate fraction. Conversely, water that is bonded to chemical structures on the surfaces of particles will not be freely exchangeable, and hence will be included with the intra-particulate fraction. For these reasons, grinding reduces the amount of intra-particulate water by destroying cellular structures, but also reduces the amount of extra-particulate water as the small particles created occupy space between larger particles (Auffret, Ralet, Guillon, Barry, & Thibault, 1994).

Whilst bacterial toxins and deconjugated bile acids may be bound by chemical or physical interactions with the matrices of dietary fibres (Florén & Nilsson, 1982; Robertson & Eastwood, 1981b), it has been suggested that a proportion of these substances may be sequestered in intra-particulate water (Story & Kritchevsky, 1976). Lignin-rich fibres were shown to adsorb more intra-particulate water at a given equilibrium humidity than fibres with a high cellulose content (Hill, Norton, & Newman, 2008). Furthermore, comparisons of absorption/desorption isotherms of fibrous particles from a number of different sources (Hill, Norton, & Newman, 2008) suggested that water binding capacity was directly related to the proportion of lignin. They hypothesised that the crystallinity of cellulose rich particles lowers their capacity for binding intra-particulate water, and that lignin disrupted this structure forming micro-pores that were able to sequester water. Hence, the density of such pores and the characteristics of the surfaces within and between the particles, appear to be the principal drivers that affect the water holding capacity and binding of various solutes (Eastwood & Morri, 1992; Zuman, Ainso, Paden, & Pethica, 1988).

To date, little work has been conducted to quantify the relative proportions of the extra-particulate and intra-particulate components of water that associate with the finely milled particles commonly used in commercially formulated foods. In this work we quantify the relative proportions of extra- and intra-particulate water in a range of fibrous particles that exhibit differing degrees of lignification and that are of a size distribution commonly used in food ingredients. We also examine the effect of simulated gastric and small intestinal digestion of the particles on the relative proportions of these water fractions and compare them with those recovered from human faeces and pig digesta.

2. Materials and methods

2.1. Fibre particles

Two commercially available preparations of plant fibres 'WF600' (J. Rettenmaier & Söhne, Rosenberg, Germany) and "Prolux' (Oppenheimer Pty Ltd., NSW, Australia), that were derived from the wheat plant (possibly the straw) and that are commonly used in food products, were used as examples of particles with a high cellulose content. A finely milled food grade wood fibre (Lignocel® Type C120, J. Rettenmaier & Söhne, Rosenberg, Germany) derived from a species of *Pinus* and supplied by Plant and Food Research Ltd, New Zealand, was comprised of particles with higher lignin content. Kellogg's AllBran®, a breakfast supplement, was representative of a food product containing large fibre particles. The proportions of cellulose, hemicellulose and lignin in each of these materials were determined according to Robertson and Van Soest (1981).

Particulate fractions were isolated from the faeces of a volunteer human subject maintained for three days on a dietary supplement of 100 g per day of AllBran®. The particulate fractions were harvested from the colons of five randomly selected pigs immediately after slaughter at an abattoir. Samples of human faeces and pig digesta were stored at −20 °C pending use. In both cases 20 g (wwb) aliquots of sample were thoroughly mixed prior to passing through a 1 mm sieve. Each filtrate was washed in deionised water before being used in subsequent analyses.

The commercial fibre particles 'as supplied', contained significant amounts of moisture, typically between 6% and 8%, while fibre recovered from the gut contents was saturated. As removal of this water by drying at 108 °C but not 40 °C caused irreversible changes in their ability to reabsorb water, all volumetric analyses were conducted on the undried 'as supplied' fibre and where drying was required, (at 40 °C). Separate aliquots of all particle types were dried in a forced draft oven at 108 °C to constant weight for about 2 h to determine their 'absolute' moisture content. The water removed by drying at 108 °C was tightly bound. When this water was removed from the fibre particles the volume of the fibres determined by pycnometry was reduced by 6.7%. Given the low water activity of the 'as supplied' fibres (Aw = 0.4–0.6) and the high temperature required to remove this water, it is likely that the tightly bound water component in the 'as supplied' fibre is not available for the sequestration of gut solutes.

2.2. In vitro digestion of particles

Aliquots of the four commercial fibre particles were subjected to *in vitro* digestion that simulated gastric and small intestinal digestion using the method of Mishra, Monro, & Hedderley (2008) with slight modification. All reagents used for this section were supplied by Merck and were of analytical grade. Fifty grams of raw commercial fibre particles were dispersed in 250 ml of deionised water at 37 °C and the pH adjusted to 2.5 (±0.2) with 1 M HCl (approximately 0.5 ml). Then 10 ml of 10% (w/v) porcine pepsin (P7000, Sigma–Aldrich, USA) dissolved in 0.05 M HCl was added to the mixture and stirred at 130 rpm for 30 min at 37 °C. The pH remained around 2.5 throughout the period of digestion. Twenty millilitres of a 1 M solution of $NaHCO_3$ was then added followed by 50 ml of 0.1 M Na maleate buffer at pH 6.0 containing 0.02% (w/v) sodium azide and 1 mM $CaCl_2$, after which 5 ml of 2.5% porcine bile extract (B8631, Sigma–Aldrich, USA) and 50 ml of 1.0% (w/v) of porcine pancreatin (P7545, Sigma–Aldrich, USA) dissolved in 0.1 M sodium maleate buffer at pH 6 and 1 ml of a 3260 U/ml (soluble starch) solution of amyloglucosidase (Megazyme International, Ireland) dissolved in water were added. The volume of digestate was subsequently made up to 550 ml with distilled water and the mixture stirred for 2 h at 37 °C. The fibre particles were allowed to settle for 2 h and the supernatant was then decanted. The particles were subsequently re-suspended in distilled water and again decanted, this procedure being repeated six times before the settled *in vitro* digested fibre particles were collected and dried at 40 °C for 12 h before storage for further use.