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Interactions between cellulose ethers and a bile salt in the control of lipid digestion of lipid-based systems

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ABSTRACT

In order to gain new insights into the potential of specific dietary fibres to control lipid digestion, the goal of this work is to study the main interactions between commercial cellulose ethers, as dietary fibre, and a bile salt, as an important duodenal component present during the digestibility of lipids. These interactions have been evaluated in two different scenarios found for an oil-in-water emulsion on its transit through the duodenum. Namely, interactions in the continuous phase and competitive adsorption at the oil–water interface have been looked at by means of micro-differential scanning calorimetry (micro-DSC) and interfacial tension (IT). Micro-DSC revealed that the presence of the bile salt affects the thermogelation process of cellulose derivatives, suggesting binding to cellulose ethers. The effect on thermogelation seems to be cellulose type-dependent. IT measurements proved the ability of cellulose ethers to compete for the oil–water interface in the presence of the bile salt. Interactions in the bulk might have an impact on this interfacial scenario. These findings may have implications in the digestion of emulsified lipids, hence providing a springboard to develop new cellulose-based food products with improved functional properties.

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1. Introduction

Cellulose derivatives belong to a wide group called dietary fibres which have been shown to provide health benefits in the diet by, for instance, lowering blood cholesterol (Anderson & Siesel, 1990; Kritchevsky & Story, 1993). This is due to the ability of certain types of dietary fibre to interfere with the process of digestion in a number of different and related ways. On the one hand, dietary fibres bind bile salts in the duodenum which are sequestered and eventually excreted (Story, Furumoto, & Buhman, 1997). Hence, dietary fibres reduce bile re-absorption, inducing the synthesis of bile salts from blood cholesterol to restore the content lost (Lee, Kim, & Kim, 1999; Zarras & Vogl, 1999). On the other hand, an alternative mechanism proposed to explain the reduction of blood cholesterol levels by dietary fibre is the prevention of lipid absorption (Jenkins, Kendall, & Ransom, 1998; Lairon, 1996; Yokoyama et al., 2011), which can be partially related to the sequestration of bile salt due to binding. Indeed, the binding mechanism may influence lipid absorption by affecting the process of lipid digestion. This is because bile salts play a crucial role in lipid digestion within the duodenum (upper small intestine), where the majority of this process occurs, *ca.* 70–90% (Fave, Coste, & Armand, 2004). When lipids eventually reach the small intestine, they are normally present as small lipid droplets dispersed in a compositionally and structurally complex aqueous medium. Bile salts are secreted into the duodenum and adsorb onto the surface of lipid droplets to further emulsify them and to prepare this interface for the enzymatic breakdown by the pancreatic lipase. This enzyme anchors to the lipid–water interface with the help of its cofactor colipase, previously adsorbed on the bile salt-covered interface, by forming a lipase–colipase complex (Reis, Holmberg, Watzke, Leser, & Miller, 2009). Lipase then hydrolyses the lipids (lipolysis), mainly composed of triglycerides, into free fatty acids, monoglycerides and diglycerides. Some of these products are soluble, so that they can be removed from the surface of lipid droplets and become incorporated within micelles/vesicles of bile salts in order to be absorbed by the intestinal mucosa.

Therefore, any way whereby dietary fibre can interfere with the above process may also affect the rate of lipid digestion and hence absorption. Several possible mechanisms that may alter this process include the binding of dietary fibre to bile salts or to the enzymes, the adsorption of dietary fibre on the oil–water interface, forming a protective layer against the action of bile salts and lipase, and intraluminal disruption of mixed micelles of digested lipids and bile salts, reducing transport into the mucosal surface barrier (Vahouny et al., 1988).

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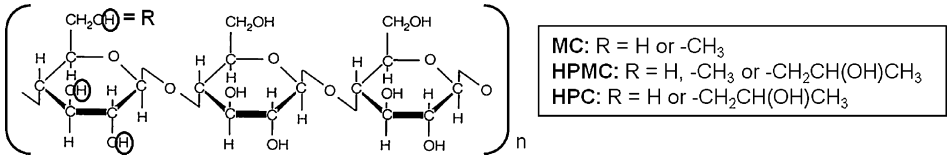
Table 1
Physicochemical characteristics of the cellulose ethers (Sarkar, 1979).

Cellulose ether		$DS_{methoxyl}$	$MS_{hydroxypropyl}$	Viscosity range (mPa·s)	M_w range (kDa)
MC	A4C	1.8	0	400 (2 wt%)	120–150
	A4M			4000 (2 wt%)	300–500
HPMC	K4M	1.4	0.21	4000 (2 wt%)	300–500
HPC	HF	0	4	1500–3000 (1 wt%)	1115



Despite the known ability of dietary fibres to influence lipid absorption, to our best knowledge there is a need of fundamental studies on their potential to control lipid digestion (Beysseriat, Decker, & McClements, 2006; Tokle, Lesmes, Decker, & McClements, 2012). Previous work in literature reported on electrostatic interactions between chitosan and a bile salt (Thongngam & McClements, 2005), dynamic molecular contact of beta-glucan with a bile salt and entrapment of bile micelles by an arabinoxylan matrix without direct molecular interaction (Gunness, Flanagan, & Gidley, 2010). However, the mechanisms by which dietary fibres interact with bile salts in the digestive tract are still not known. Hence, a systematic approach should be taken to identify the dominant mechanism for each specific type of dietary fibre to influence lipid digestion, in order to better understand the underlying molecular effects of digestion conditions in real complex food emulsions containing specific dietary fibres.

A previous study on the interactions between synthetic polymeric surfactants and bile salts in oil-in-water emulsions has shown that binding of these polymers to a bile salt may have an impact on the interfacial properties of the emulsions related to access for digestion (Torcello-Gómez, Maldonado-Valderrama, Jódar-Reyes, & Foster, 2013). Cellulose derivatives offer a good candidate mirroring the block-copolymer research. For that reason, the aim of this work is to study the main interactions between cellulose derivatives, as non-ionic dietary fibre, and bile salts, as an important duodenal component. Specifically, we have focused on the interactions in the aqueous phase and competitive adsorption at the oil–water interface as scenarios of oil-in-water emulsions as they pass through the duodenum. These interactions have been characterised in the aqueous phase by means of micro-differential scanning calorimetry, and at the oil–water interface through competitive adsorption by means of interfacial tension measurements. This combination of techniques has shown to be successful in previous work (Torcello-Gómez et al., 2013).

Commercial cellulose ethers have been chosen as dietary fibre due to the highly functional properties which are important in the manufacture process of food, such as: surface activity, binding, thickening, and a wide range of viscosities. For the cellulose derivatives chosen, the native cellulose backbone has been chemically modified by partially reacting the hydroxyl groups in each sugar ring (Table 1) to provide the following cellulose ethers: methylcellulose (MC), in which methyl group is the sole substituent, hydroxypropylmethylcellulose (HPMC) in which methyl group remains the dominant substituent, but incorporating smaller amounts of the larger and more polar hydroxypropyl group, and hydroxypropylcellulose (HPC) where the hydroxypropyl group is the sole substituent. These are three of the four cellulose derivative forms used in the food area for their surface tension reducing properties (Mezdour, Cuvelier, Cash, & Michon, 2007). This selection allows us to discuss the results comparing their molecular properties, such as the molecular weight in the case of MC, and the type and number of substituents for a set of cellulose ethers within the same range of viscosity (MC, HPMC and HPC) and hence molecular weight (see Table 1), since the viscosity of aqueous solutions of cellulose ethers is proportional to its molecular weight. Sodium taurodeoxycholate (NaTDC) was used as an example of a typical bile salt, since bile salts behave in a qualitative similar manner despite their large variety (Maldonado-Valderrama, Wilde, Macierzanka, & Mackie, 2011).

Beginning to quantify the dynamics of interactions in the bulk and the impact that has on the interfacial properties, related to access for digestion, provides new insights for designing rules for application. These new findings can be exploited in tailoring both novel food and pharmacological matrices with improved functional properties.

2. Materials and methods

2.1. Materials

METHOCEL™ A4C, A4M and K4M from the Dow Chemical Company were used without purification. The initial letter 'A' denotes MC and 'K' corresponds to HPMC. In each case, '4C' and '4M' denote a solution viscosity of 400 and 4000 mPa·s, respectively, measured at 2 wt% concentration under standard conditions in a capillary viscometer. Klucel® HPC HF was purchased from Hercules. The different levels of incorporation of the two substituents (degree of substitution, DS, and molar substitution, MS) and range of viscosities are indicated in Table 1.

The bile salt used in this study is sodium taurodeoxycholate (NaTDC, 97% purity) from Sigma-Aldrich. It is negatively charged at pH 7 and its molecular weight is 521.7 g/mol.

The aqueous phase was 1.13 mM phosphate buffer (pH 7) prepared with ultrapure water purified in a Pur1te Select system.

Highly refined olive oil was also purchased from Sigma-Aldrich, and purified with activated magnesium silicate (Florisil®, Fluka) to eliminate free fatty acids and surface active impurities. Namely, a mixture of oil and Florisil® in proportion 2:1 wt/wt was shaken mildly for 3 h and centrifuged at 4000 rpm for 30 min in a bench centrifuge. It was then filtered through Whatman filter paper #1 under vacuum and stored away from light.

2.2. Sample preparation

Cellulose ethers stock solutions (2 wt%) were prepared as follows. Approximately one-third of the required final volume of aqueous phase was heated to ~80 °C, and then cellulose ether powder was added carefully under stirring. The complete solubilisation was obtained by adding the remaining aqueous phase at room temperature and continuing the agitation of the solution for at least 2 h at room temperature. It was then stored at 4 °C overnight to achieve the maximum hydration, and without stirring to eliminate air bubbles. Aliquots from the stock solutions were dried at 50 °C

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