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# Emulsification Speed of Amphiphilic Starches

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## INTRODUCTION

Amphiphilic starches (sodium octenylsuccinate starches) are a well-known emulsifier for various food and pharma applications. Typical applications are the encapsulation of lipophilic compounds such as vitamins and other micronutrients or the direct or indirect solubilization of drug substances [1].

To obtain the emulsifying activity, the hydrophilic starch molecule undergoes a chemical modification, introducing the lipophilic octenylsuccinate group. The obtained anionic starch ester is hydrophilic and acts as emulsifier (type o/w) at low pH values. Due to numerous application cases and related requests on the emulsifier properties, it is common to customize commercial products by modifying the starch backbone. This could be done e.g. by enzymatic treatment, dextrinification or thinning with mineral acids. This modification of the starch structure modulates crucial application properties such as the micellization in aqueous solution and the adsorption properties to hydrophobic surfaces. Literature reports, that the structure of the starch chain is of major importance of the emulsification capacity [2] and the emulsion structure.

## MATERIALS AND METHODS

### Materials

Several amphiphilic starches were used. They differ in the industrial way to design the starch backbone structure:

- CLEARGUM® CO 03, batch E957B (Roquette Frères, France) obtained by using  $\alpha$ -amylase.
- HI-CAP® 100 (Ingredion Inc.) obtained by using  $\beta$ -amylase.
- CLEARGUM® CO A1, batch E6060 (Roquette Frères, France) designed by using a dextrinification step.

Spray dried glucose syrup Glucidex® 29 (Roquette Frères, France), Medium chain triglycerides and sunflower oil were of laboratory / food quality.

### Methods

**Emulsification trials** : GLUCIDEX® 29 was dissolved (200 g in 350 g demineralized water per test) to form a stock solution. The requested starch content (typically 75 g or 150 g) is added to 550 g of the stock solution and dissolved under moderate agitation to obtain a clear solution. The final starch concentration in the solutions (without oil) was 12% and 21.5% respectively.

Just before the emulsification trials, the needed oil quantity is added. The emulsion is done with a Polytron® (Kinematika AG Switzerland) equipment. Samples were taken after 2 min, 4 min, 8 min and 12 minutes homogenization for an immediate measurement of the droplet size with a Malvern 3000 equipment. Some samples have been kept for 24 hours at 40°C before a new analysis to estimate the emulsion stability.

**Molecular weight** : analyzed in aqueous solution via size exclusion chromatography, equipped with a MALS detector.

**Critical aggregation concentration**: the surface tensions (Krüss K12 equipment with Wilhelmy plate) of aqueous solutions are plotted against the starch concentration (in logarithmic scale).

## RESULTS AND DISCUSSION

Many commercial amphiphilic starches need long time and energy input to form a stable fine emulsion.

Figure 1 illustrates a typical evolution of the droplet size using a starch of the dextrin type.

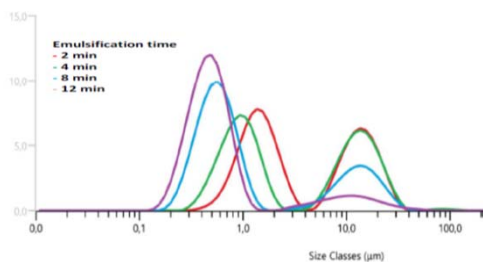


Figure 1. The emulsification speed of system using CLEARGUM® CO A1 as emulsifier (with 150 g starch and 150 ml MCT).

Surprisingly, we have observed a significantly faster emulsification when using enzymatically treated ( $\alpha$ - or  $\beta$ -amylase) products. Fine emulsions are forming with enzymatically treated starches without going through a bimodal phase. Figure 2 demonstrates the efficiency of these products in highly oil loaded systems.

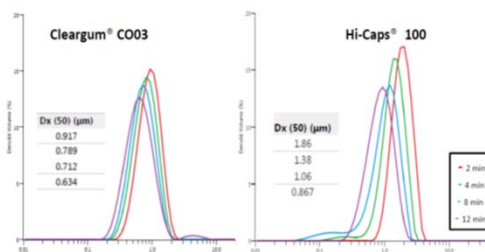


Figure 2. The emulsification speeds of CLEARGUM CO 03 or HI-CAP 100 (with 150 g starch and 350 ml MCT)

Such starches do not contain *de nature* other bonds such as the  $\alpha$ -1,4 and  $\alpha$ -1,6 glucosidic bonds, unlike starches of the dextrin type. The slow emulsification speed of thermally treated starches might be caused by the steric hindrance of the starch molecules to form a hydrocolloid multilayer around the oil droplets [3]. Other physical parameter can't explain the faster action of enzymatic treated starches. CLEARGUM CO 03 has approximately a 4-fold higher molecular weight compared with CLEARGUM® CO A1 (200 kDa vs. 50 kDa). The critical aggregation concentration has been found to be very similar with 1.5% for CLEARAM CO A1 and 3.0% for CLEARGUM® CO 03.

Both enzymatic treated starches (with  $\alpha$ - or  $\beta$ -amylase) turned out to be efficient and rapid emulsifier. In all tested application cases, fine emulsions are forming without going through a bimodal phase.

All preparations with MCT (up to 350 g) and all emulsions with sunflower oil up to the 250 g were stable for 24h at 40°C. No evolution of the droplet size could be measured, indicating a beginning coalescence of these emulsions.

Considering the speed to emulsify as sole application parameter, the best product was CLEARGUM® CO 03. Under all test conditions and with different oils it resulted in finer droplet size under similar conditions. Table 1 illustrates some examples.

Recipe	Droplet size [µm] after homogenisation		
	4min	12 min	
150g Hi-Caps	+ 250g SF oil	1,34	0,80
	+ 250g MCT	1,25	0,90
150g Cleargum CO03	+ 250g SF oil	0,90	0,80
	+ 250g MCT	0,80	0,62

Table 1. Comparative droplet size in emulsification trials with enzymatically treated amphiphilic starches

## CONCLUSION

The molecular starch fine structure modifies significantly the emulsification efficiency of amphiphilic starch ester. It seems, that rather linear starch derivatives, consisting of  $\alpha$ -1,4 &  $\alpha$ -1,6 glucosidic bonds arrange more rapidly around oil drops to stabilize an emulsion.

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