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Crystallisation kinetics of ultra-pure lactose

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ABSTRACT
An ultra-pure lactose feed was produced from a patented process (Durham et al. 1997) and shown to be free of lactose phosphate (LP) and other impurities. The objectives of this work were to establish the crystallisation kinetics of the ultra-pure lactose during cooling seeded batch crystallisations and to compare the results with similar data obtained for commercial pharmaceutical grade α-lactose (PGα-L). Based on experimental data, a nonlinear least squares dynamic optimisation technique was used to define the growth rate constant K of a power law function. For all conditions studied, K was higher for the ultra pure feed than for the commercial feed. The use of the ultra pure feed, free of LP, produced smaller crystals with a narrow particle size distribution and in higher yield.

INTRODUCTION
A previous study established the kinetics of isothermal seeded batch crystallisation of ultra-pure lactose in water, in the presence of LP (Lifran et al. 2005). This study presents the results of seeded batch cooling crystallisations of both ultra-pure lactose and PGα-L contaminated with LP. The results confirmed LP inhibition on a population of crystals growing in similar conditions to the industrial lactose crystallisation.

MATERIAL AND METHODS
Ultra-pure lactose produced by a zero-waste process (Durham et al. 1997) was shown to be free of LP compared to PGα-L. Commercial pharmaceutical grade alpha-lactose monohydrate 100 mesh was from The Lactose Company of New Zealand (Lactose NZ Ltd, Hawera, New Zealand). Seeded-batch cooling crystallisations were performed with different cooling strategies developed by Vu et al. (2004). Crystallisations were conducted in 2L continuously stirred glass vessels, using 1g seeds/100g dissolved lactose. Samples were collected every half an hour, or at longer intervals, over 26 hours. Samples (2 x 1.5ml) were analysed for solid content, particle median size and solution concentration. The growth rate G ($\mu m.hr^{-1}$) was correlated with α-lactose concentration C_α and solubility $C_{\alpha s}$ (g α-lactose/100g water), in the following equation: $G = KT(C_\alpha - C_{\alpha s})^n$ where K is the growth rate constant, T the temperature (°C) and n the power of the driving force of the crystallisation. Values of the constant K were found by solving a nonlinear least squares dynamic optimisation problem, in MATLAB, to fit the model equations to the measured variables.

RESULTS AND DISCUSSION
Fixing n at 2.8, the value previously obtained for ultra pure lactose (Lifran et al. 2005), different values of K were found for ultra-pure and commercial lactose feeds. The growth rate constant K was higher for the ultra pure feed than for the commercial feed (Table 1). For a crystallisation period of 12 hours, the yield was significantly reduced while crystal size decreased and particle size distribution was broadened for the commercial lactose. A large overshoot was observed (Figure 1) for the median size whereas it was not present for the control. After 26 hours, the differences with the control were reduced for the median size; however using ultra pure lactose provided 28% greater crystal recovery than for the commercial lactose. The maximum solid content achievable for a feed contaminated with LP,

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under the conditions used, was 0.20 g lactose /g slurry after 26 hours. The maximum practical crystal content in an industrial batch crystalliser is around 0.25 g/g. The use of the ultra pure feed, free of LP, allowed this value to be reached in 12 hours. After 26 hours, the crystals from the ultra-pure feed were smaller and still in higher amount than for the commercial feed (Table 2).

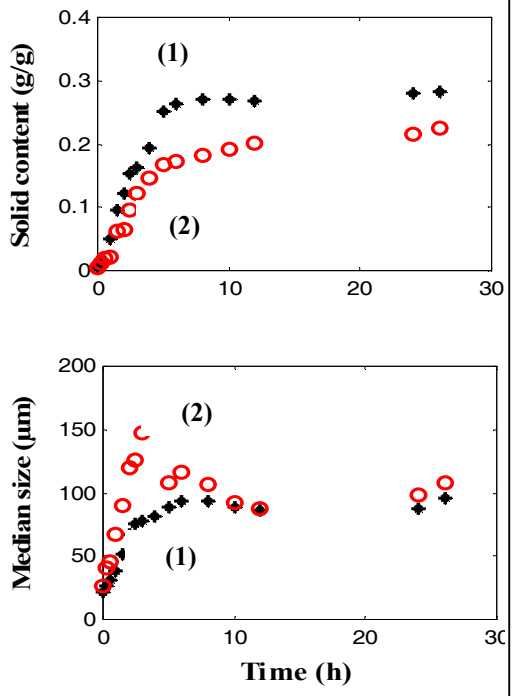

Table 1. Growth rate constant K of ultra-pure and commercial lactose preparations.

Feed type	K x 10^4 (µm/h)/(g α-lactose/100g water)$^{2.8}$
Ultra-pure	0.21
Commercial	0.0019

Table 2. Crystallisation performances of the two studied feeds after 26 hours.

Feed type	Median size (µm)	Yield (g crystals/ g dissolved lactose)
Ultra-pure	95	73
Commercial	127	46

Figure 1. . Evolution of solid content and median size with time. (1): ultra-pure lactose, (2): commercial lactose.

CONCLUSION

During seeded-batch cooling crystallisations, an ultra-pure lactose feed, free of LP, crystallised faster and gave smaller crystals in higher yield compared to commercial lactose.

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