Influence of the Operating Conditions on Acid Degumming Process in Sunflower Seed Oil

Sir:

In a recent paper (1) we studied the kinetic extraction of phospholipids present in crude sunflower oils, such as phosphatidylcholine (PC), phosphatidylethanolamine (PE), phosphatidylinositol (PI), phosphatidylserine (PS) and phosphatidic acid (PA), for monitoring the efficiency of the degumming process under conditions used by the oil industry. We have now extended this study, investigating the effect of different variables related mainly to the acid refining step (type and concentration of degumming agents, temperature and contact time), in accordance with technological requirements. Diverse degumming agents [phosphoric acid, citric acid, and acid mixtures (phosphoric/citric acids, 50:50), all used in a 2.5% ratio (degumming agent/oil) at a concentration of 8, 10, and 13%], temperatures (60, 70, and 80°C), contact times (5–35 min with moderate agitation) were used. With the methodology described in Reference 1, the high-performance liquid chromatography–evaporative light scattering detection profiles and the residual phosphorus content were determined, applying the statistical analysis of data (2). The residual total phospholipid content in supernatant oils degummed at 60°C/0% measured the least at 35 min for citric and phosphoric acid and at 25 min for the acid mixture ($P < 0.05$). At 70°C/10%, citric acid exhibited a notable superiority as a degumming agent, with the lowest total phospholipid content at 25 min ($P < 0.05$); similar results were obtained at 35 min for the acid mixture and for phosphoric acid. The lowest remaining phospholipid content was observed at 35 (acid mixture) and 15 min (citric and phosphoric acid) at 80°C/10%. In the phosphoric acid treatment, the residual content was greater ($P < 0.05$) than with the other agents assayed (Fig. 1).

For the acid mixture, at 60 and 70°C and 10% degumming agent we registered a rapid elimination of the remnant content of PC, PA, and PS, followed by PI. PE was the most difficult compound to remove. At 80°C, a rapid decrease in the contents of PC, PA and PI was detected; PE was extracted more slowly (data not shown).

The effect of degumming agent at 8%, 70°C presented the least efficient treatment in the case of phosphoric acid; citric acid and the acid mixture were more effective (Fig. 2A). PE, PI, and PS were the most difficult compounds to remove. Figure 2B shows the differential behavior for the treatment with phosphoric acid.

At 13%, lowest total phospholipid content was achieved at 25 min for citric acid and for acid mixture; equivalent levels in 15 min with phosphoric acid ($P < 0.05$) (Fig. 3A). Also, we registered the lowest remnant amount of PE at 25 min, followed by PI and PS (Fig. 3B). For citric acid, in all cases, the evolution of total phospholipid content was in agreement with the residual phosphorous content profiles.

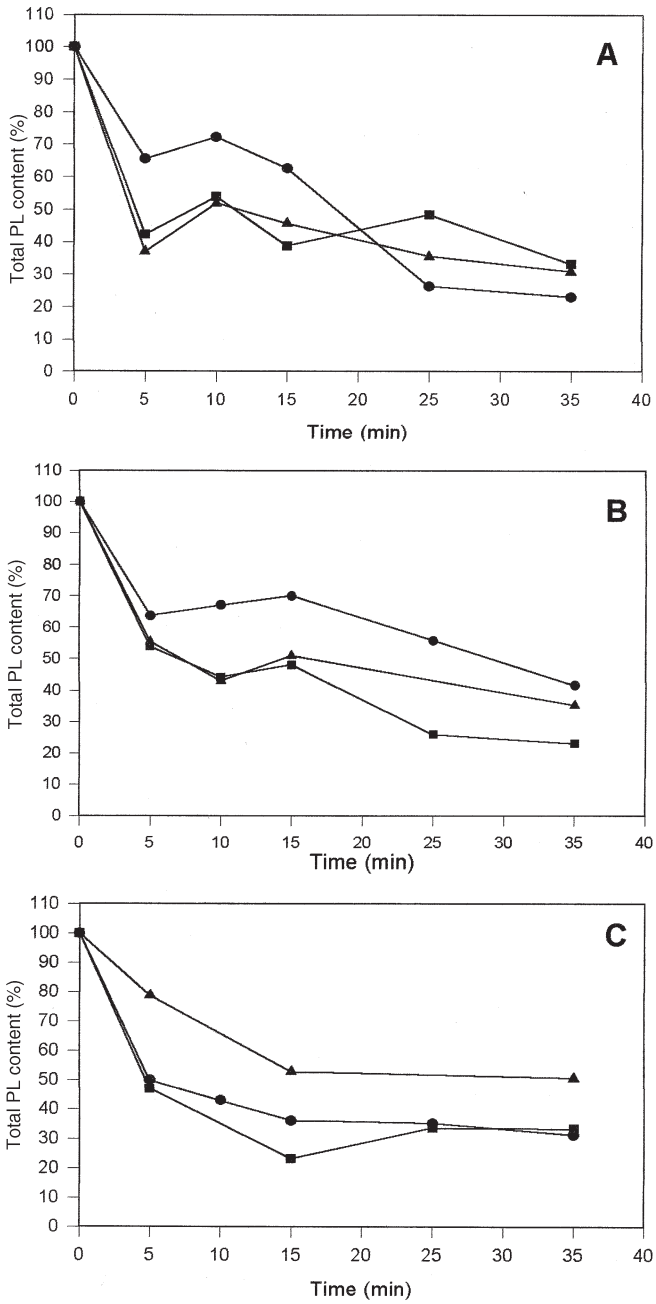

FIG. 1. Kinetic evolution of the total phospholipid (PL) content in acid-degummed oils, 2.5% ratio (degumming agent/oil). ▲ = phosphoric acid, 10%; ■ = citric acid, 10%; ● = acid mixture, 10% (phosphoric acid/citric acid, 50:50). Total PL content (%) = total PL content referred to $t = 0$ min. (A) T = 60°C, (B) T = 70°C, (C) T = 80°C.

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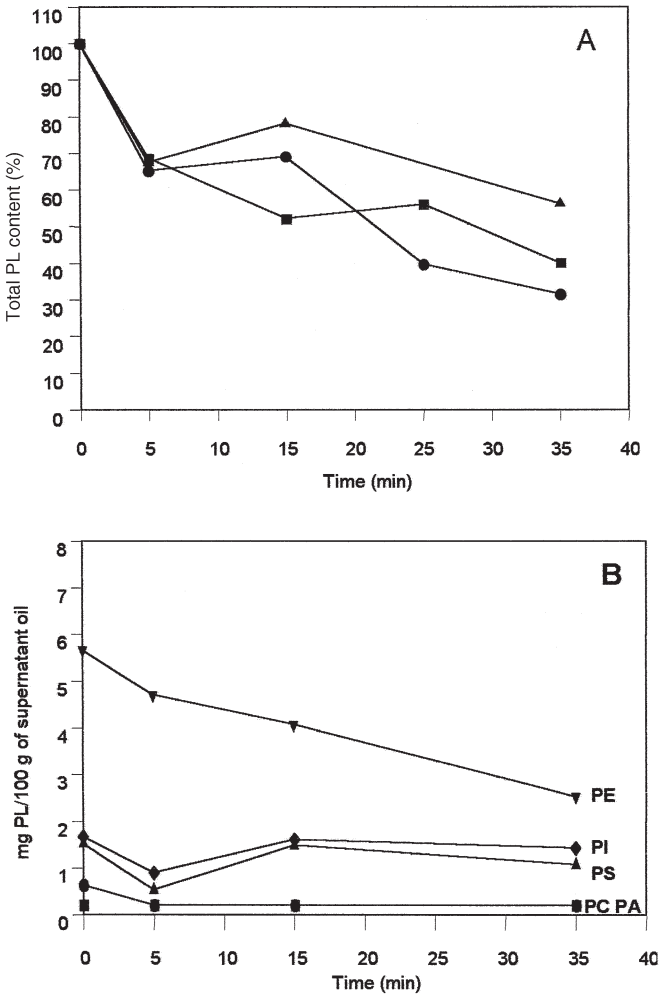

FIG. 2. (A) Kinetic evolution of the total PL content in acid degummed oils, 2.5% ratio (degumming agent 8%/oil). ▲ = phosphoric acid; ■ = citric acid; ● = acid mixture (phosphoric acid/citric acid, 50:50). Total PL content (%) = total PL content referred to $t = 0$ min. (B) Kinetic evolution of the PL content in acid degummed oils 2.5% ratio (degumming agent/oil), T = 70°C, phosphoric acid 8%. ● = phosphatidylcholine (PC); ■ = phosphatidic acid (PA); ▲ = phosphatidylserine (PS); ▼ = phosphatidylethanolamine (PE); ◆ = phosphatidylinositol (PI). Plotted values are the mean of three determinations. See Figure 1 for other abbreviation.

The optimal conditions for degumming with phosphoric acid were 60–70°C, 35 min (removal: 70–75%); for citric acid, 70°C–25 min, and for acid mixtures (77%), 60°C–25 min ($P < 0.05$). The increase from 70 to 80ºC did not represent an advantage when 10% citric acid was used. Similar effects were registered with an increase up to 13%. The best extraction levels were: citric acid 10%; phosphoric acid 10%; acid mixture 8% ($P < 0.05$). Citric acid would be a good alternative compatible with the environment. This information provides a good approach for the improvement of the degumming process.

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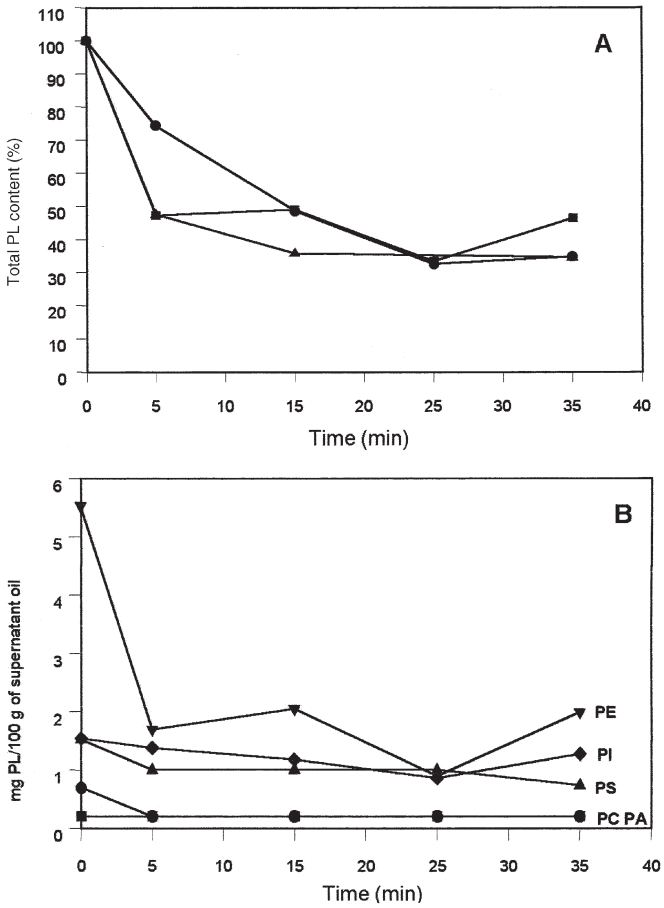

FIG. 3. (A) Kinetic evolution of total PL content in acid degummed oils, 2.5% ratio (degumming agent 13%/oil) T = 70°C, ▲ = PA; ■ = citric acid; ● = acid mixture (phosphoric acid/citric acid, 50:50). Total PL content (%) = total PL content referred to $t = 0$ min. (B) Kinetic evolution of the PL content in acid degummed oils 2.5% ratio (degumming agent/oil) T = 70°C, citric acid 13%. ● = PC; ■ = PA; ▲ = PS; ▼ = PE; ◆ = PI. Plotted values are the mean of three determinations. For abbreviations see Figures 1 and 2.

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