

Pilot scale enrichment of polyphenols from natural extract by CPC/CCC for cosmetic uses

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Nowadays the cosmetic industry uses natural extracts with high a content of active compounds for their formulations. Presented here is the purification and scale up by CPC of a natural plant extract to obtain high quantities of an enriched polyphenols mixture.

The objective was first to develop a CPC method to enrich two major compounds in a crude mixture [Fig. 1]. The scale up was then done from the Armen SCPC-250 system to the Armen SCPC-12,5L system. Three injections of 50, 100 and 150g were carried out and the fractions obtained were then analyzed by HPLC for control and cross checking.

Recovery need to be over 35% (w/w) and total area of target compounds need to be over 95 % in HPLC

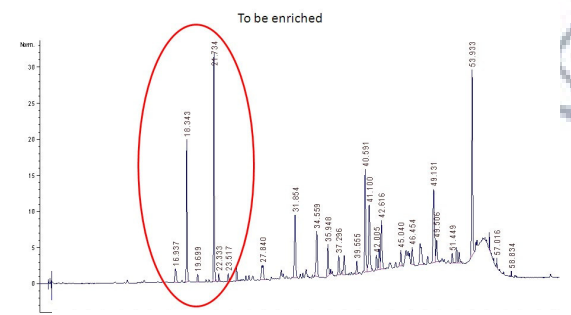


Figure 1 : Analytical HPLC chromatogram of crude mixture

Keywords: cosmetic, scale up, polyphenol, CPC/CCC

Lab scale enrichment

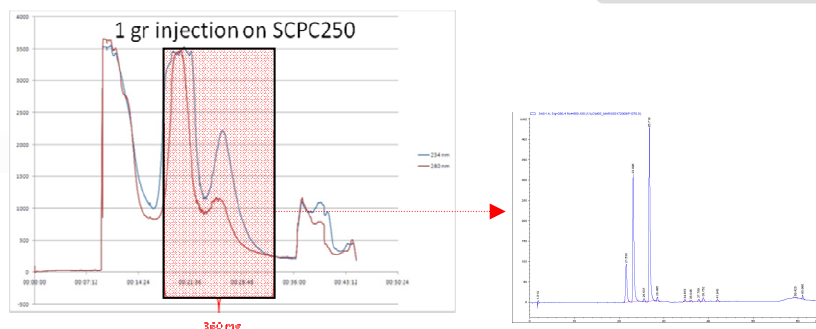


Figure 4 : SCPC chromatogram of 1 gr injection on SCPC-250 and HPLC analysis of enriched fraction

Quantity injected	Run time	Solvent consumption	Enriched fraction weigh	Yield	HPLC control ¹
1 gr	45 mn	1L	360 mg	36%	95%

Table 1 : results from the 1 gr injection on SCPC250

¹ : total % area of targets compounds

The method was first developed on a lab scale instrument Armen SCPC-250 couple with a Armen Spot prep II system [Fig. 2]. All automatically pilot by Armen Glider CPC Software : Stationary phase loading, equilibration, injection, elution, extrusion [Fig. 3].

A 1 gr injection of the crude mixture [Fig. 1] was done with a n-BuOH/AcOEt/TFA 0.1% system in ascending mode (upper organic phase as mobile), the flow rate is fixed at 10 ml/mn and rotation speed at 2000 rpm during 32 mn. An extrusion from 32 to 45 mn at 30 ml/mn allows rinsing the entire column. 1L of solvent was use during this 45 mn run. 360 mg of highly enriched mixture were recovered for a 36% yield (w/w) and analysed by HPLC [Fig. 4]. The fraction obtained is conform to the requirement [Tab. 1]

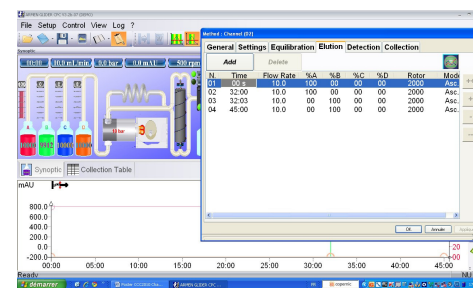


Figure 3 : Method developed on Armen Glider CPC software for automation



Figure 2 : SCPC250 from Armen coupled with spot prep II

Scale up to pilot scale



Figure 5 : Complete SCPC-12,5 L from Armen with 1.5L/mn pump

Quantity injected	Run time ¹	Solvent consumption (L/gr injected)	Enriched fraction weigh	Yield	HPLC control ²
50 gr	70 mn	1L	21,4 gr	43%	93%
100 gr	80 mn	0.5L	39,1 gr	40,5%	94%
150 gr	70 mn	0.33L	56,9gr	35,3%	89%

Table 2 : results from the 50, 100 and 150 gr injections on SCPC12,5

¹ : without extrusion

² : % area of target compounds

The scale up was done on a complete pilot scale instrument Armen SCPC-12.5L equipped with 2X6.25L CPC column 1,5L/mn pump, all automatically pilot by Armen Glider Software [Fig. 5].

The volume ratio between the SCPC-250 and a SCPC-12.5L column is X50. Consequently, it was decided to inject 50 gr for the first run on the SCPC-12.5L [Fig. 6] Yield obtained were better than on lab scale run [tab.2] so two runs of 100 [fig.7] and 150 gr [Fig. 8] were done for optimization. Each enriched fraction were analysed by HPLC.

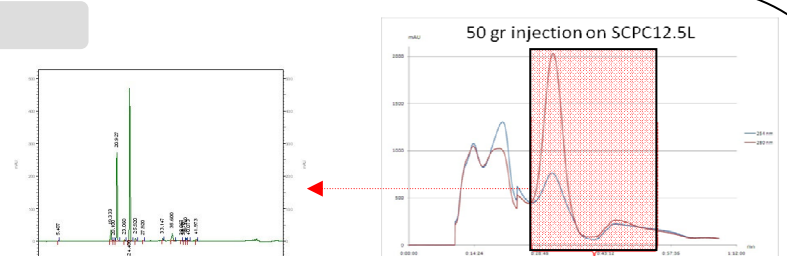


Figure 6 : CPC chromatogram of 50 gr injection on SCPC-12.5L and HPLC analysis of enriched fraction

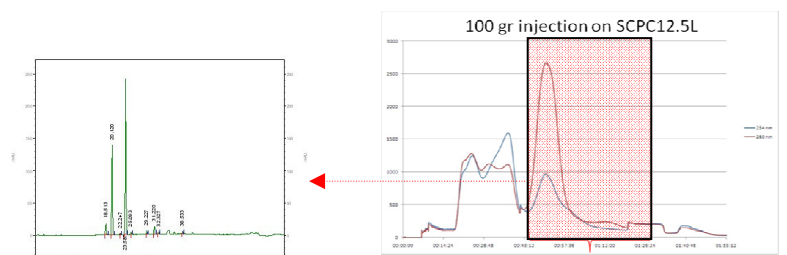


Figure 7 : CPC chromatogram of 100 gr injection on SCPC-12.5L and HPLC analysis of enriched fraction

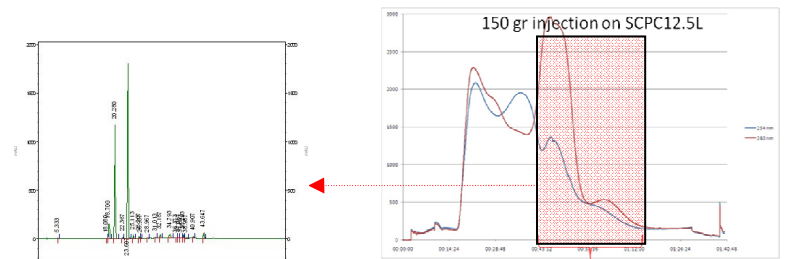


Figure 8 : CPC chromatogram of 150 gr injection on SCPC-12.5L and HPLC analysis of enriched fraction

Conclusion

This study shows a direct scale up from a lab scale CPC system equipped with a 250 ml column to a pilot scale system with a 12,5L column. Optimisation of quantity injected is done on the pilot scale system to increase productivity and reduce solvent consumption. 150 gr injection in this case is the limit but still match the requirement of the project.