

Contents

Chapter 1	Uses and Applications of Extracts from Natural Sources	1
	<i>R. N. Cavalcanti, T. Forster-Carneiro, M. T. M. S. Gomes, M. A. Rostagno, J. M. Prado and M. A. A. Meireles</i>	
1.1	Introduction	1
1.2	Uses and Applications	3
1.2.1	Coloring Agents	4
1.2.2	Flavors and Fragrances: Essential Oils	19
1.2.3	Edible Fats and Oils	31
1.2.4	Functional Foods and Nutraceuticals	36
1.3	Conclusions	46
	Acknowledgements	46
	References	46
Chapter 2	Extraction of Natural Products: Principles and Fundamental Aspects	58
	<i>M. Palma, G. F. Barbero, Z. Piñeiro, A. Liazid, C. G. Barroso, M. A. Rostagno, J. M. Prado and M. A. A. Meireles</i>	
2.1	Introduction	58
2.2	Principles and Fundamentals of Extraction	59
2.3	Exhaustive Versus Non-exhaustive Extraction	
	Methods	66
2.4	Conventional Extraction Techniques	67
2.4.1	Soaking	67
2.4.2	Soxhlet	69
2.4.3	Distillation with Water and/or Steam	73

2.5	Main Extraction Variables	78
2.5.1	Preparation of the Solid	78
2.5.2	Solvent	79
2.5.3	Temperature	82
2.5.4	Time	82
2.5.5	Solvent to Feed Ratio	82
2.6	Case Study	83
2.7	Conclusions	85
	Acknowledgements	86
	References	86
Chapter 3	Ultrasound-assisted Extraction	89
	<i>Daniella Pingret, Anne-Sylvie Fabiano-Tixier and Farid Chemat</i>	
3.1	Introduction	89
3.2	Ultrasound-assisted Extraction	90
3.2.1	Ultrasound Principles	90
3.2.2	Instrumentation	93
3.2.3	Important Parameters	96
3.2.4	Ultrasound-assisted Extraction: Applications in Food	102
3.3	Examples of Solvent-free Ultrasound-assisted Extraction of Carotenoids	105
3.3.1	Carotenoids Uses and Conventional Extraction	105
3.3.2	Solvent-free Ultrasound-assisted Extraction of β -Carotene	106
3.3.3	Analysis and Evaluation of UAE Process	107
3.4	Costs and Investment in Industrial Ultrasound	108
3.5	Conclusion	108
	References	109
Chapter 4	Microwave-assisted Extraction	113
	<i>Emilie Destandau, Thomas Michel and Claire Elfakir</i>	
4.1	Introduction	113
4.2	Principles of Microwave-assisted Extraction	114
4.2.1	Microwave Heating Principle	114
4.2.2	Microwave Heating Applied to Plant Matrices	117
4.3	Microwave Instrumentation	118
4.3.1	Oven Design	119
4.3.2	Reactor Design	120

4.4	Parameter Influence on Microwave-assisted Extraction	121
4.4.1	Solvent	122
4.4.2	Temperature and Pressure	125
4.4.3	Extraction Time	126
4.4.4	Power	126
4.4.5	Nature of the Matrix	127
4.5	Trends in Microwave-assisted Extraction and Applications	128
4.5.1	Extraction of Sensitive Compounds	128
4.5.2	Extraction Methods Improved by Microwave Heating	130
4.5.3	Green Extraction without Solvent	135
4.6	Case Study	144
4.6.1	Optimization of the Pressurized Solvent-free Microwave Extraction (PSFME) Procedure	144
4.6.2	Influence of the Number of Cycles	145
4.6.3	Proposed Mechanism of PSFME	147
4.6.4	Comparison with other Extraction Methods	148
4.6.5	Advantages of PSFME	150
4.7	Conclusion	150
	List of Abbreviations	152
	References	152

Chapter 5 Accelerated Liquid Extraction 157

Feliciano Priego-Capote and María del Pilar Delgado de la Torre

5.1	Introduction	157
5.2	Static Accelerated Solvent Extraction (Static ASE)	158
5.2.1	Steps Involved in the Static ASE Process	158
5.2.2	Static ASE Commercial and Laboratory-designed Devices	161
5.3	Dynamic Accelerated Solvent Extraction (Dynamic ASE)	163
5.3.1	Steps Involved in the Dynamic ASE Process	163
5.3.2	Dynamic ASE Laboratory-designed Devices	164
5.4	Coupling ASE to Other Steps of the Analytical Process	165
5.5	Parameters Affecting Performance in ASE	167
5.5.1	Temperature	167
5.5.2	Pressure	169
5.5.3	Type of Solvent	169
5.5.4	Solvent to Feed Ratio	170
5.5.5	Sample Composition	171

5.5.6	Particle Size	171
5.5.7	Extraction Time	171
5.6	Comparison of ASE with other Extraction Techniques	172
5.7	Applications of ASE for the Isolation of Natural Products	176
5.7.1	Lipids	182
5.7.2	Volatile Compounds	183
5.7.3	Polar Compounds	184
5.8	Case Study	187
5.8.1	Optimisation of the Main Variables Involved in SHLE	187
5.8.2	Influence of Extraction pH	189
5.8.3	Comparison of SHLE with MAE and UAE for Extraction of Vine Shoots	189
5.9	Conclusions: Benefits and Limitations of ASE for Isolation of Natural Products	190
	Acknowledgements	190
	References	190

Chapter 6 Supercritical Fluid Extraction 196

Jose A. Mendiola, Miguel Herrero, María Castro-Puyana and Elena Ibáñez

6.1	Introduction	196
6.2	Fundamentals of Supercritical Fluid Extraction	197
6.2.1	Physical Properties of Supercritical Fluids	197
6.2.2	Supercritical Solvents	199
6.3	Instrumentation	201
6.4	Parameters Affecting the Extraction Process	203
6.4.1	Raw Material (Particle Size, Porosity, Location of the Solute, Moisture Content)	204
6.4.2	Solubility (Pressure and Temperature)	205
6.4.3	Use of Modifiers	208
6.4.4	Solvent Flow Rate (Solvent-to-Feed Ratio)	209
6.5	Applications	209
6.5.1	Plants	209
6.5.2	Marine Products	213
6.5.3	Agricultural and Food By-products	216
6.6	Case Study	220
6.6.1	Effect of Extraction Time	220
6.6.2	Effect of Pressure, Temperature and Modifier	221
6.6.3	Effect of Solvent	222
6.7	Future Trends and Conclusions	223
	References	225

Chapter 7	Recent Trends and Perspectives for the Extraction of Natural Products	231
	<i>M. E. M. Braga, I. J. Seabra, A. M. A. Dias and H. C. de Sousa</i>	
7.1	Introduction	231
7.2	Target Extracts/Compounds	236
7.3	Raw Materials	244
7.4	Extraction Methods	250
7.4.1	Microwave-assisted Extraction	253
7.4.2	Ultrasound-assisted Extraction	255
7.4.3	High-pressure Liquid Extraction	257
7.4.4	Supercritical Fluid Extraction	258
7.5	Extraction Solvents and Solvent Mixtures	261
7.5.1	Extraction Solvent Modification with Additives (Enzymes, H^+ / OH^- , Surfactants)	263
7.5.2	Solvent Mixtures and Non-conventional Highly Hydrophobic Organic Solvents	267
7.5.3	Ionic Liquids	268
7.5.4	Aqueous Biphasic Systems (ABS)	269
7.5.5	Tunable Solvents	271
7.6	Conclusions and Future Perspectives	274
	References	275
Chapter 8	Post-extraction Processes: Improvement of Functional Characteristics of Extracts	285
	<i>Ángel Martín, Soraya Rodríguez-Rojo, Alexander Navarrete, Esther de Paz, Joao Queiroz and María José Cocero</i>	
8.1	Introduction	285
8.2	Purification of Extracts and Elimination of Solvents	286
8.2.1	Evaporation of Solvents	287
8.2.2	Freeze-drying	287
8.2.3	Reverse Osmosis	287
8.3	Particle Size Reduction	289
8.3.1	Top-down Methods	290
8.3.2	Bottom-up Methods	291
8.4	Formulation	298
8.4.1	Solvent Evaporation Method	300
8.4.2	Spray-drying Technique	301
8.4.3	High-pressure Emulsion Techniques	301
8.4.4	Supercritical Fluid Processes	303
8.4.5	Overview	305

8.5	Case Study: Formulation of β -carotene as a Natural Colorant	305
8.5.1	Formulation of β -carotene by Precipitation from Pressurized Organic Solvent-on-water Emulsions	307
8.5.2	Formulation of β -carotene with Soybean Lecithin by PGSS-drying	309
8.5.3	Co-precipitation of β -carotene with Polyethylene Glycol by Supercritical Anti-solvent Process (SAS)	309
8.5.4	Formulation of β -carotene by Supercritical Extraction from an Emulsion (SEE)	310
8.6	Conclusions	311
	References	311
Chapter 9	Isolation and Purification of Natural Products	314
	<i>Wang Xiao, Fang Lei, Zhao Hengqiang and Lin Xiaojing</i>	
9.1	Introduction	314
9.2	Pre-isolation or Enrichment	315
9.2.1	Solvent Partitioning	316
9.2.2	Adsorption Enrichment	318
9.2.3	Membrane Separation	318
9.2.4	Solid Phase Extraction (SPE)	321
9.3	Purification	323
9.3.1	Chromatographic Techniques	323
9.3.2	Crystallization	339
9.4	Case Studies	340
9.4.1	Isolation of Saponins from <i>Clematis chinensis</i>	340
9.4.2	Isolation of Tritoniopsins A–D from <i>Cladiella krempfi</i>	341
9.4.3	Isolation of <i>cis</i> -Clerodane-type Furanoditerpenoids from <i>Tinospora crispa</i>	341
9.4.4	Isolation of Flavonoids from <i>Paeonia suffruticosa</i>	344
9.4.5	Isolation of Alkaloids from <i>Stephania kwangsiensis</i>	347
9.4.6	Isolation of Psoralen and Isopsoralen from <i>Psoralea corylitolia</i>	348
9.4.7	Isolation of Six Isoflavones from <i>Semen sojae praeparatum</i> by Prep-HPLC	348

9.4.8	Isolation of Anthocyanins from Eggplant	352
9.4.9	Isolation and Purification of Flavonoid and Isoflavonoid from <i>Sophora japonica</i>	354
9.5	Conclusions	356
	References	357

Chapter 10 Scale-up of Extraction Processes 363

Julian Martínez and Luiz Paulo Sales Silva

10.1	Introduction	363
10.2	Fundamental Aspects of Scale-up Operations	364
10.2.1	What is Scale-up?	364
10.2.2	Scale-up Criteria	366
10.3	Factors Involved	372
10.3.1	Solubility	373
10.3.2	Solvent Flow Rate	374
10.3.3	Substrate Properties	374
10.3.4	Extraction Bed Geometry	375
10.4	State of the Art	376
10.4.1	Models for Extraction Processes	376
10.4.2	Some Examples of Scale-up Criteria in Extraction Processes	380
10.4.3	Scale-up Correlations	387
10.4.4	Configurations of Industrial Units	388
10.4.5	Some Published Works on Scale-up of Extraction Processes	390
10.5	Case Study: Supercritical CO ₂ Extraction from Red Pepper	391
10.5.1	Experimental Procedures	391
10.5.2	Results and Discussion	393
10.6	Conclusion	396
	References	397

Chapter 11 Integration of Pressurized Fluid-based Technologies for Natural Product Processing 399

Diego T. Santos, Maria T. M. S. Gomes, Renata Vardanega, Mauricio A. Rostagno and M. Angela A. Meireles

11.1	Introduction	399
11.2	Sequential Extraction using Different Process Conditions or Techniques	400
11.3	On-line Fractionation/Purification	404
11.3.1	On-line Separators: Fractionation by Changes in Temperature and Pressure	404

11.3.2	On-line Extraction and Adsorptive Purification Processes	406
11.3.3	On-line Coupling of Extraction and Membrane Processes for Purification	418
11.4	Integration of Pressurized Fluids to Different Technologies for Extract Stabilization	420
11.5	Case Study – Integrated Extraction and Encapsulation of Bixin from Annato Seeds	425
11.5.1	Materials and Methods	425
11.5.2	Results and Discussion	430
11.6	Conclusions	437
	Acknowledgements	438
	References	438
Chapter 12	Economic Evaluation of Natural Product Extraction Processes	442
	<i>Camila G. Pereira, Juliana M. Prado and M. Angela A. Meireles</i>	
12.1	Introduction	442
12.2	Cost Estimation of Industrial Processes	443
12.2.1	Costs Associated with the Raw Material	444
12.2.2	Costs Associated with the Operational Conditions	445
12.2.3	Costs Associated with the Industrial Requirements	445
12.3	Cost Estimation Procedures	446
12.3.1	Cost Estimate as a Function of Equipment Capacity	446
12.3.2	Lang Factor	448
12.3.3	Manufacturing Cost Estimation	448
12.4	Manufacturing Cost of Vegetable Extracts	450
12.4.1	Supercritical Extraction Process	450
12.4.2	Other Extraction Processes	464
12.5	Case Study	465
12.5.1	Introduction	465
12.5.2	Materials and Methods	465
12.5.3	Results and Discussion	466
12.6	Conclusion	469
	Acknowledgement	469
	References	469
	Subject Index	472