TABLE OF CONTENTS

LIST OF ACRONYMS 27

INTRODUCTION 29

1. Biodegradable active materials for food packaging 30

2. Active packaging materials 36

3. Use of electrospinning to develop active films 42

3.1. Description of the technique 42

3.2. Encapsulation of active compounds by electrospinning and development of multilayer active films 44

4. References 54

OBJECTIVES 63

CHAPTERS 65

I. 1 Carvacrol encapsulation in starch or PCL based matrices by electrospinning 69

Abstract 71

1. Introduction 72

2. Materials and methods 75

2.1. Raw materials and reagents 75

2.2. Preparation of formulations 75

2.3. Characterization of the initial solution/dispersion properties 76

2.4. Electrospinning process 77

2.5. Characterization of electrospun products 78

2.5.1. Nanostructure of electrospun material 78

2.5.2. Encapsulating efficiency of CA 78

2.6. Statistical analysis 79

3. Results and discussion 79

3.1. Properties of the carvacrol liquid formulations 79

3.2. Characterization of the electrospun product 87

3.3. CA encapsulation efficiency 90

4. Conclusions 94

5. Acknowledgments 95

6. References 95

I. 2 Release kinetics and antimicrobial properties of carvacrol encapsulated in electrospun poly-(ε-caprolactone) nanofibres. Application in starch multilayer films. 101

Abstract 103

1. Introduction 104

2. Materials and methods 106

2.1. Materials and reagents 106

2.2. Obtaining and characterizing the electrospun fibre of CA-loaded PCL 106

2.3. Release kinetics of CA in different food simulants 107

2.3.1 CA release mathematical modelling 108

2.3.2. Prediction of CA antimicrobial effect based on the release study 109

2.4. Incorporation of PCL fibres in multilayer starch films 109

2.5. Thermal analysis 110

2.6. Antimicrobial properties 111

2.7. Statistical analysis 111

3. Results and discussion 112

3.1. Electrospun fibre layers of CA loaded PCL 112

4. Conclusions 129

5. Acknowledgements 129

6. References 130

I. 3 Biodegradation of thermoplastic starch films containing electrospun poly-(ε-caprolactone) encapsulating carvacrol 137

Abstract 139

1. Introduction 140

2. Materials and experimental design 141

2.1. Materials 141

2.2. Films preparation 142

2.3. Samples characterization 142

2.4. Compost and synthetic solid residue (SSR) 143
2.5. Disintegration test 144
2.6. Biodegradation test 144
2.7. Statistical analysis 145
3. Results 146
3.1. Properties of multilayer films 146
3.2. Compost characteristics 146
3.3. Disintegration test 148
3.4. Biodegradation test 153
4. Conclusions 156
5. Acknowledgements 157
6. References 157

II. 1 Poly(vinyl alcohol)-based materials encapsulating carvacrol obtained by solvent casting and electrospinning 163
Abstract 165
1. Introduction 166
2. Materials and methods 168
2.1. Materials and reagents 168
2.2. Preparation of the liquid formulations 168
2.3. Obtaining the dry encapsulating material 169
2.4. Characterization of the liquid systems 170
2.4.1. Particle size distribution 170
2.4.2. Rheological behaviour 170
2.4.3. Conductivity, surface tension and ζ potential 170
2.5. Characterization of the solid material 171
2.5.1. Microstructure 171
2.5.2. Encapsulation efficiency 171
2.5.3. Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) 171
2.6. Statistical analysis 172

3. Results and discussion 172
3.1. Properties of the liquid systems 172
3.2. Characterization of the solid material 176
4. Conclusions 186
5. Acknowledgements 186
6. References 186

II. 2 Poly(lactic acid) based materials encapsulating carvacrol obtained by solvent casting and electrospinning 193
Abstract 195
1. Introduction 196
2. Materials and Methods 198
2.1. Materials 198
2.2. Obtaining the CA encapsulating matrices 198
2.3. Characterization of the obtained materials 199
2.3.1. Microstructure 199
2.3.2. CA encapsulating efficiency 199
2.3.3. Thermal analysis 200
2.4. Statistical analysis 200
3. Results and Discussion 201
3.1. Solvent system screening 201
3.2. Microstructure of the ES matrices 204
3.3. Carvacrol encapsulating efficiency 204
3.4. Thermal analysis 206
4. Conclusions 213
5. Acknowledgments 213
6. References 213

II. 3 Enhancement of PLA-PVA surface adhesion in bilayer assemblies by PLA aminolization 217
Abstract 219

1. Introduction 220

2. Materials and methods 222

2.1. Materials 223

2.2. Mono- and bi- layer preparation 223

2.4. Surface microstructure 227

2.5. Analysis of functional properties of bilayer films 227

2.6. Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) 228

2.7. Statistical analysis 228

3. Results and Discussion 229

3.1. Changes in the PLA surface induced by aminolization 229

3.2. Functional properties of PLA-PVA bilayer films 235

4. Conclusions 244

5. Acknowledgements 244

6. References 244

GENERAL DISCUSSION 251

CONCLUSIONS 263