## **Table of Contents**

. Intro	duction	1	1
l. Fund	amenta	als	7
1. Pla	isma osc	sillation and Mie's theory	9
1.1	Principle	es of plasma oscillation [20]	10
1.2	Scatteri	ng and absorption of small particles [23]	12
2. L(+	·)-ascorb	oic acid and its derivates	15
2.1		al properties of ascorbic acid	
		rfactants and their aggregates [28]	
		nation of gel and coagel	
		d its different structures and species	
		chloride in aqueous solutions	
3.2		es of zirconium oxide	
3.2	.1 Cry	stal structures and martensic phase transformation	27
3.2	.2 Tet	ragonal zirconia and critical crystal size	34
4. Str	uctural ir	nvestigation techniques	37
4.1	X-ray ba	ased methods	37
4.1	.1 The	e nature of X-rays [48]	37
4.1	.2 Sm	all angle X-ray scattering (SAXS)	41
4	1.1.2.1	Scattering by one electron	42
4	1.1.2.2	The scattering vector [54]	42
4	1.1.2.3	The electron density [55]	44
4	1.1.2.4	The scattering intensity [58]	45
4	.1.2.5	The auto correlation and invariant [58]	46
4	1.1.2.6	Scattering of spherical particles [58]	47
4	.1.2.7	The Guinier approximation [58]	48
4	.1.2.8	Correlation length and Porod's law of scattering [58]	49
4	.1.2.9	Scattering of particles with non-uniform electron density [58]	50
4.1	.3 X-ra	ay diffraction (XRD)	52



	4.1.3.1 Phase shift and intensity [65]	.53
	4.1.3.2 Bragg's Law of diffraction [65], [67]	.55
	4.1.3.3 The reciprocal lattice and the system of Miller indices [64], [65]	.57
	4.1.3.4 The Scherrer equation [68]	.60
4.2	Electron based method: scanning electron microscopy (SEM)	.62
4.	2.1 Principal setup [72]	
4.	2.2 The scanning process [73]	.64
III. Exp	erimental	65
5. C	hemicals	.67
5.1	Preparation of Gold Nanoparticles	.67
5.2	Preparation of zirconium-based nanoparticles	.67
6. A	nalytical Methods	.68
6.1	Thermogravimetric analysis (TGA)	.68
6.2	Differential scanning calorimetry (DSC)	.68
6.3	Bright field and phase contrast microscopy	.69
6.4	UV/Vis absorption	.70
6.5	Raman measurements	.71
6.6	Small angle X-ray scattering (SAXS)	.72
6.7	X-ray diffraction (XRD)	.73
6.8	Scanning electron microscopy (SEM)	.74
7. Sy	nthesis of L(+)ascorbyl stearate (Asc18)	.75
8. Sy	nthesis of Gold Nanoparticles	.77
8.1	Preparation with Asc18 surfactant	77
8.2	Preparation with Asc12 surfactant	78
8.3	Preparation with Asc14 Asc10 and Asc8 surfactants	79
9. Pr	eparation of ZrO <sub>2</sub> - nanoparticles	80
9.1	Synthesis of zirconium hydroxide by coprecipitation in homogeneous	
	phase (sol)	80
9.2	Preparation of hydrous zirconia gel	80

IV.Results and Discussion	83
10. Gold nanoparticles	85
10.1 Determination of the cmt of Asc18	85
10.2 Synthesized nanoparticles and their colors	86
10.2.1 Influence of reaction temperature	
10.2.2 Comparison of different concentrations	88
10.3 UV-Vis characterization	90
10.3.1 Comparison of different concentrations	90
10.3.2 Comparison of different reaction temperatures	93
10.3.2.1 Asc10	94
10.3.2.2 Asc12	95
10.3.2.3 Asc14	97
10.3.2.4 Asc18	98
10.4 SAXS characterization	101
10.4.1 The Schulz Spheres fitting model [79]	101
10.4.2 Comparison of reactions above and below the cmc	102
10.4.3 Comparison of different reaction temperatures	103
10.4.4 Comparison of AscX surfactants with different chain lengths	106
10.4.4.1 Asc12	106
10.4.4.2 Asc14	109
10.4.4.3 Asac18	111
10.5 Conclusion	113
11. 3. Zirconium hydroxide and oxide nanoparticles	115
11.1 Raman characterization	115
11.2 Dialysis of the sol and an aqueous ZrOCl <sub>2</sub> solution	117
11.2.1 Conductivity of the sol and ZrOCl <sub>2</sub> solution	
11.2.2 Progress of pH and conductivity during gel-formation	117
11.3 Characterization with microscopic methods	119
11.3.1 LM-micrographs of untreated gel	
11.3.2 LM-micrographs of squeezed gel	
11.3.3 LM-micrographs of air-dried gel	

11.3.4 LM-micrographs of collapsed gel	122
11.3.5 LM-micrographs of a freeze-dried gel	123
11.4 DSC measurements of the gel	124
11.5 Characterization by SEM	127
11.6 SAXS characterization of the gel	130
11.6.1 The unified fit model [80]-[82]	130
11.6.2 Structural parameters of the gel	132
11.7 TGA and DTG measurements	135
11.8 XRD characterization of calcined samples	139
11.8.1 Diffractogram of the gel-synthesized particles	139
11.8.2 Diffractogram of the sol-synthesized particles	140
11.8.2.1 Samples containing NaCI	141
11.8.2.2 Samples without NaCl	143
11.9 Conclusion	144
V. Annex	147
List of Figures	149
List of Tables	152
Bibliography	152