Doctoral School of Materials Sciences and Technologies
Óbuda University

HUN-REN Centre for Energy Research

Biopolymer composite for biomedical applications

Report of Third Semester

9-Junary-2024

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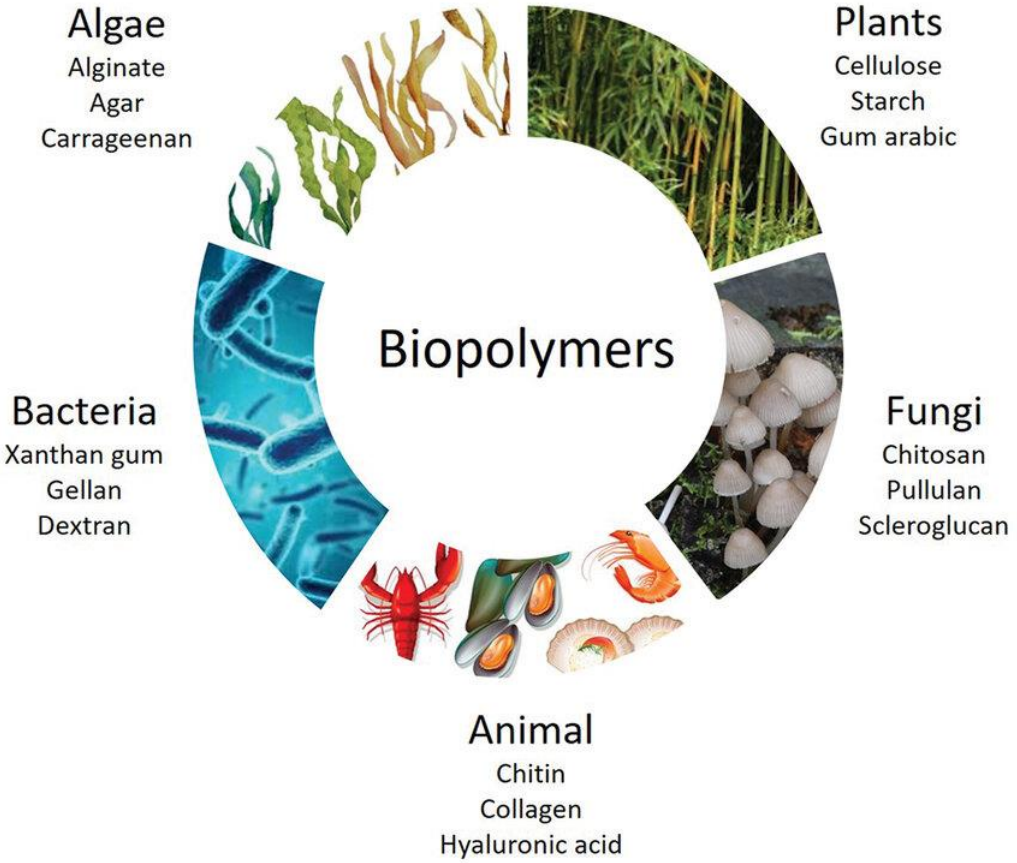
Introduction

Biopolymers

Biopolymers have been established as a promising class of materials with a wide range of applications, of which medicine stands out. Characteristics such as biocompatibility, biodegradation and non-cytotoxicity make these material excellent candidates to be used in biomedical applications

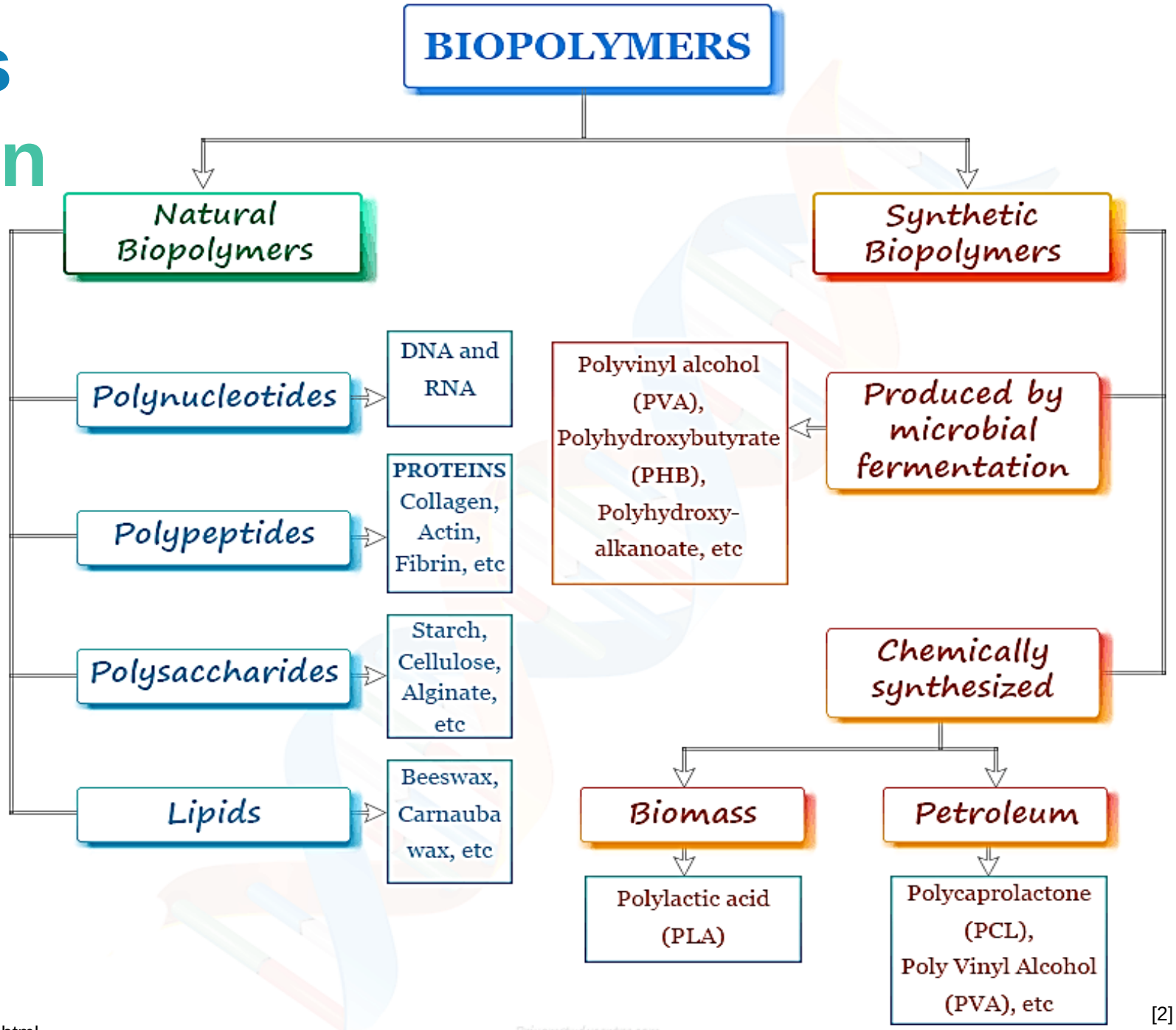
Biopolymers Sources



[1]

[1] DOI:10.1080/15440478.2023.2301364

Biopolymers
Classification

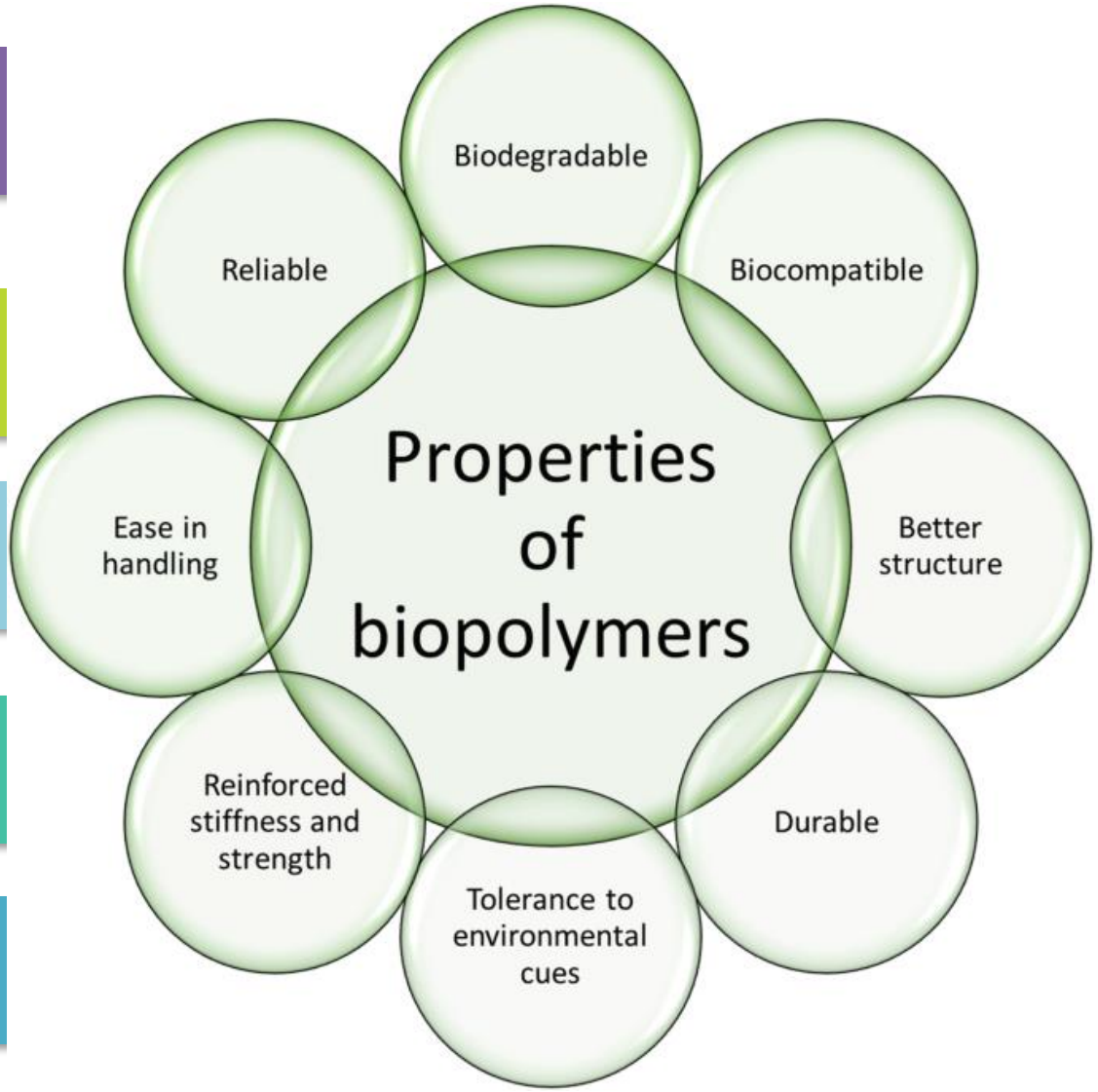


[2]

https://www.priyamstudycentre.com/2023/11/biopolymers.html

Biopolymers properties

- Superior biocompatibility
- Biodegradability
- Cell adhesion, low cost
- Proliferation
- Flexibility, easy accessibility



Biopolymers
Limitation

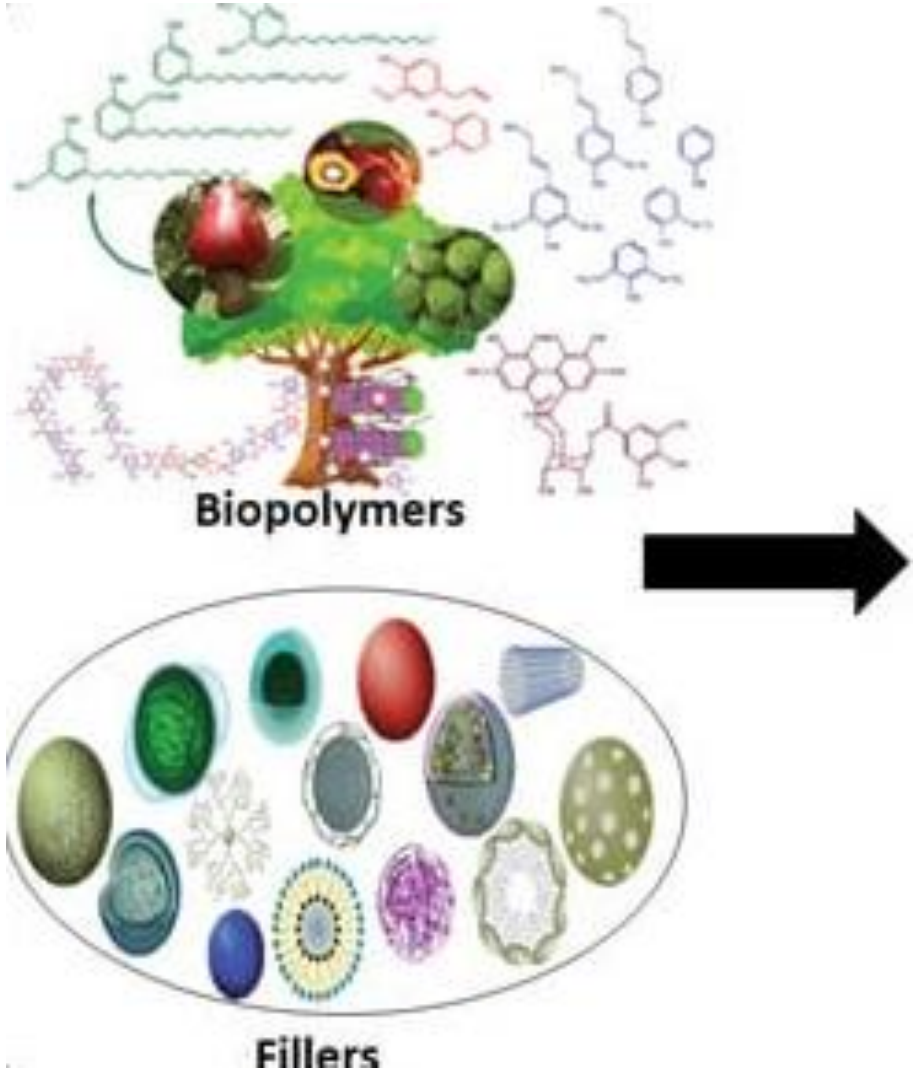
01 Poor thermal stability

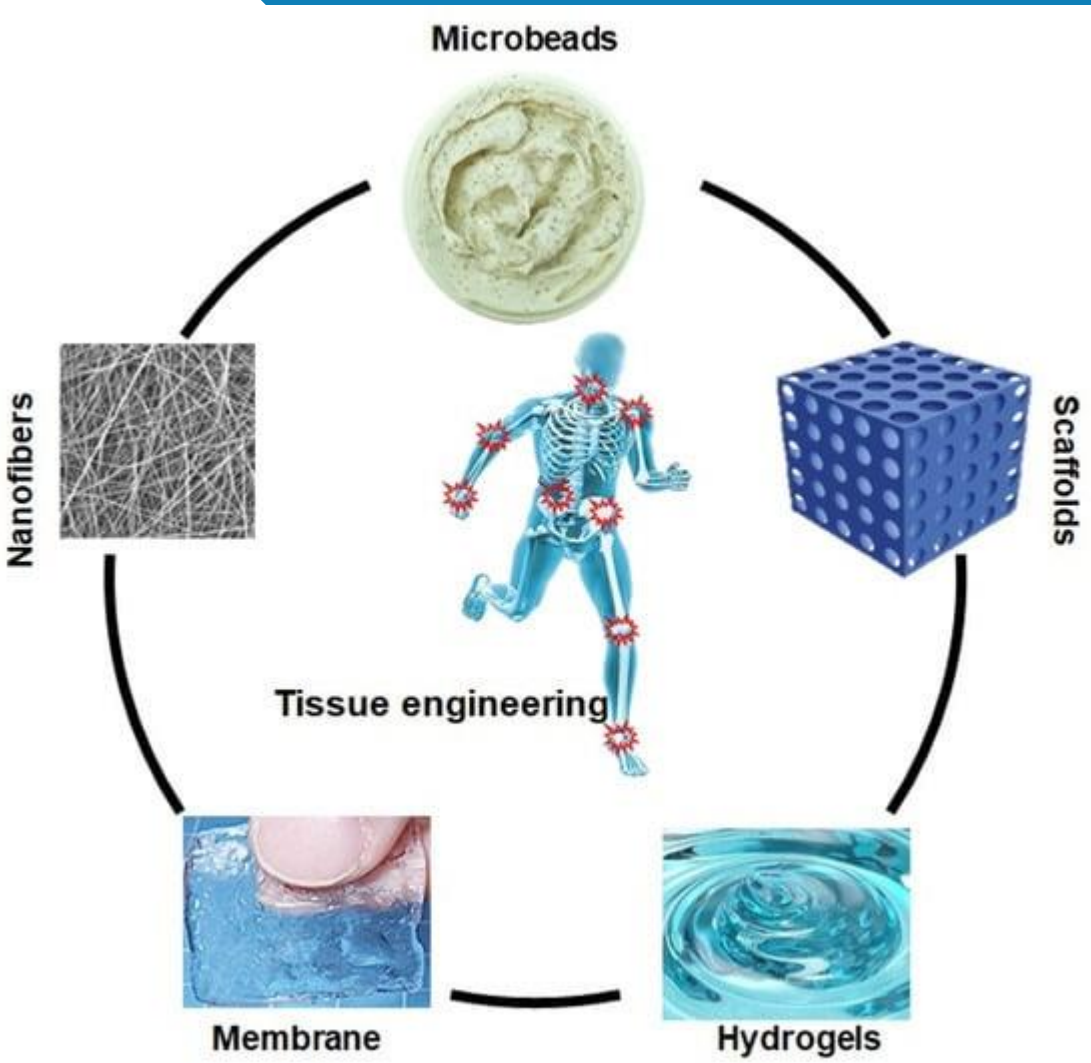
02 Poor mechanical properties

03 High degradation rate

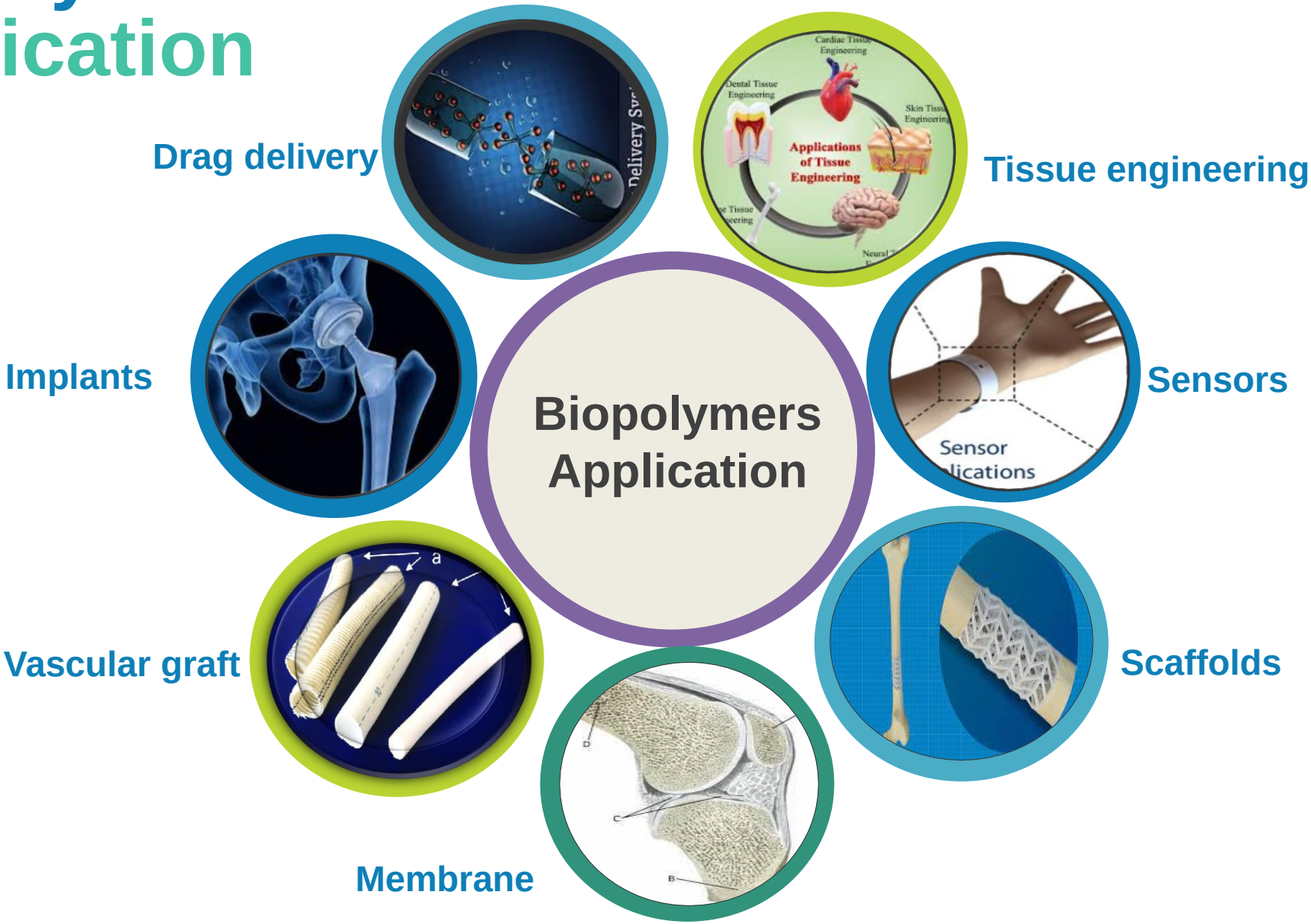
04 poor stability in an aqueous environment

Biopolymers
Composite





Biopolymers
Application



Aims and planning research

1. Preparation of CaO from natural sources (Eggshells) and study the characteristics of prepared CCaO.

2. Study the feasibility of using natural CaO as a filling material in its nano or micro-sized polymer composites by electrospinning method

3. The mechanical properties of the obtained composites are important to be studied.

4. The prepared and investigated CaO/composite materials are aimed to satisfy the requirements for biomedical applications.

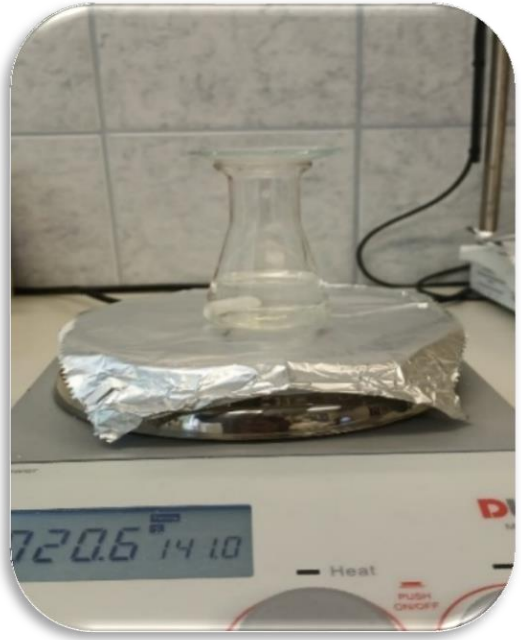
5. Characterize the CaO from eggshell and its composites:
Scanning electron microscopy (SEM), Fourier Transformed Infrared Spectroscopy (FTIR), X-ray diffraction (XRD)

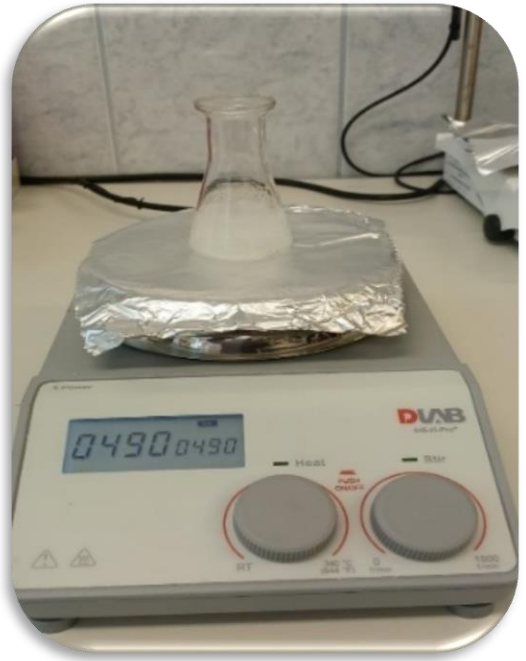
Experimental Work

Preparation of polymer by electrospinning method:

The polymer solution is prepared by mixing PVP po wder with ethyl alcohol, while CA mixing with differ ent solution using magnetic stirrer until reach to a sui table viscosity. Electrospinning techniques are used t o generate fiber through injecting the polymer soluti on via a syringe, producing a droplet at the end of th e needle and the fiber collected on collector.

polymer	Concentration %	ethanol	acetone	Acetone/ acetic acid	Acetone /DMF
PVP	10	100%	--------	------	-------
CA	(5, 10, 11, 13, 15)	--------	100%	--------	-------
CA	(5, 10,15,16,18,20)	--------	---------	70/30	-------
CA	(5, 10, 11, 13, 15)	-------	--------	-------	70/30





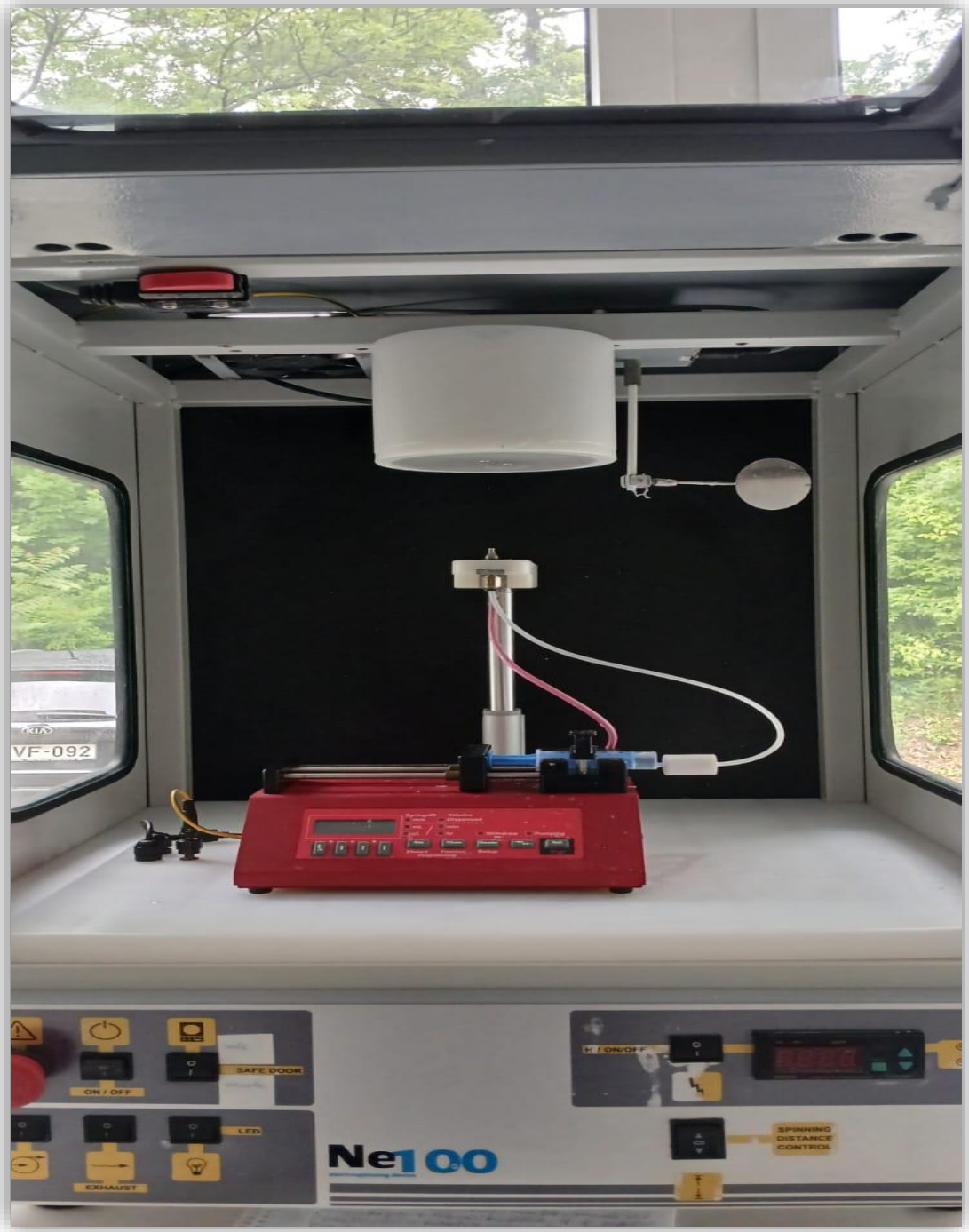
Experimental Work

There are several factors that affect the electrospinning process.

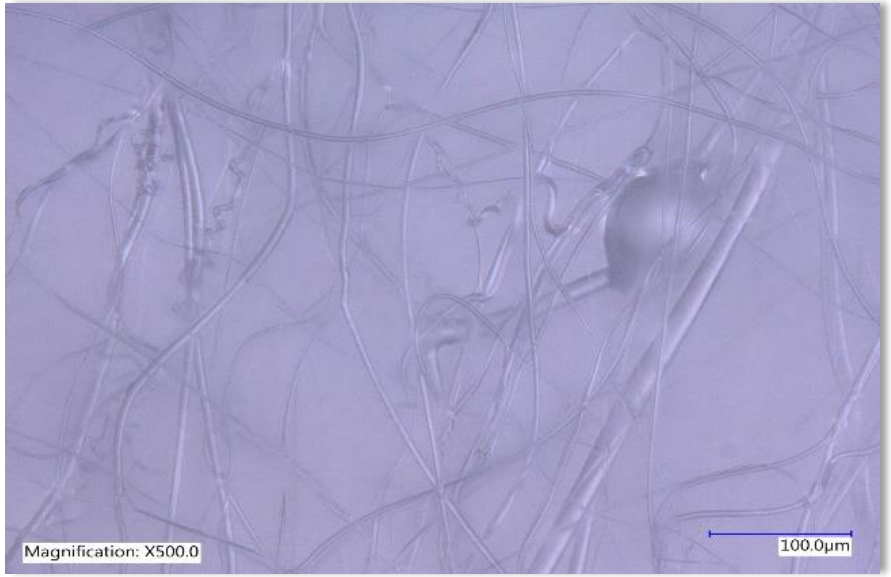
These factors are classified as electrospinning parameters, solution and environmental parameters

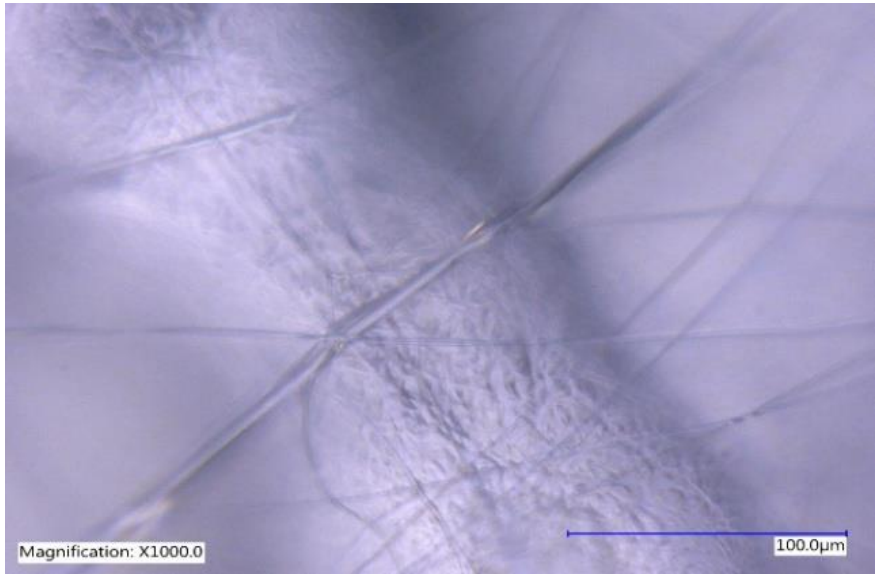
The electrospinning parameters include:

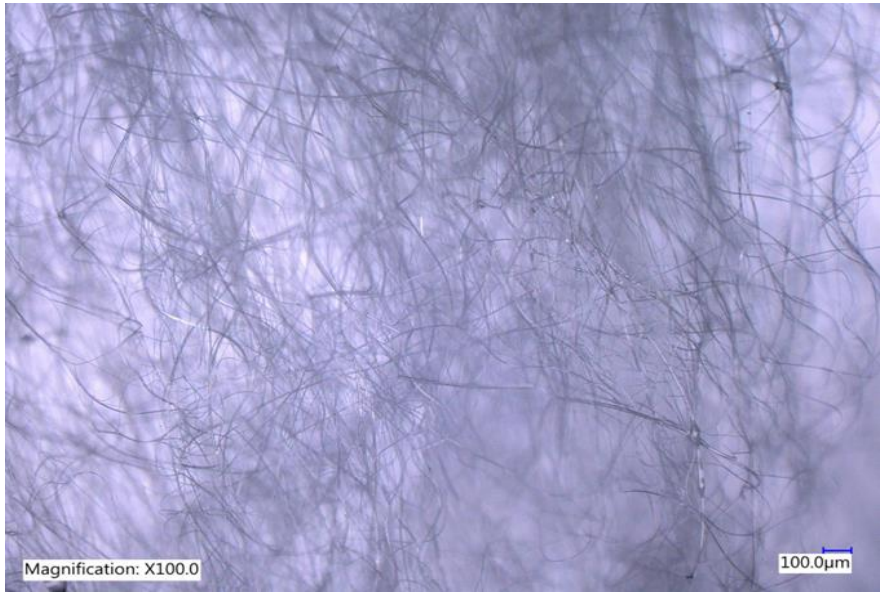
1) The applied electric field
2) Distance between the needle and collector
3) Flow rate
4) Needle diameter



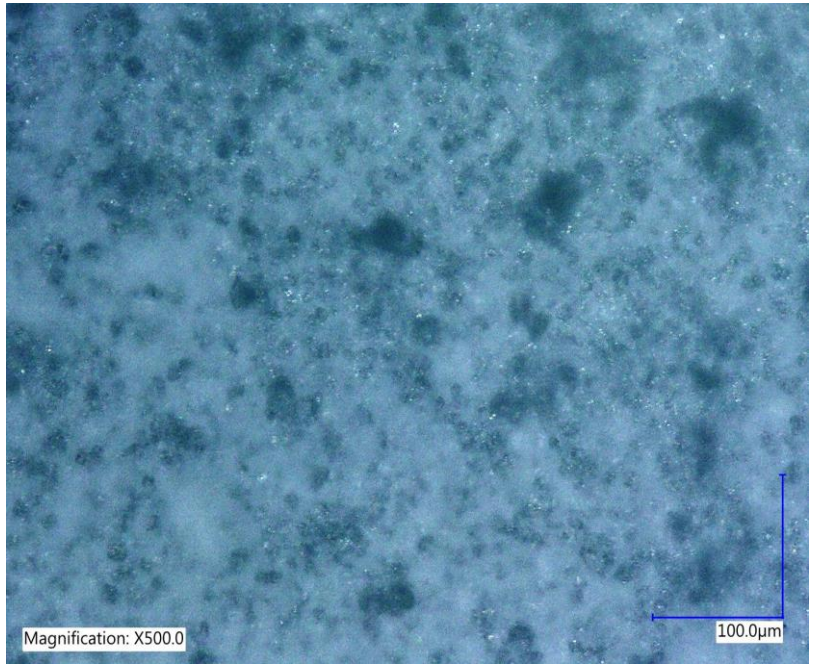
The effects of solvent system, solution concentration, the morphological appearance of pvp and cellulose acetate (CA) products were thoroughly investigated.

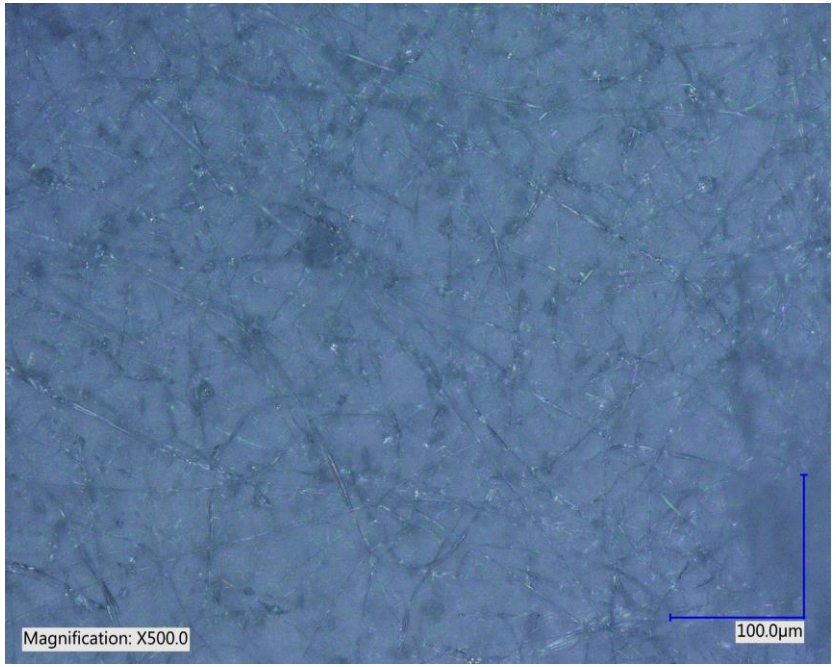


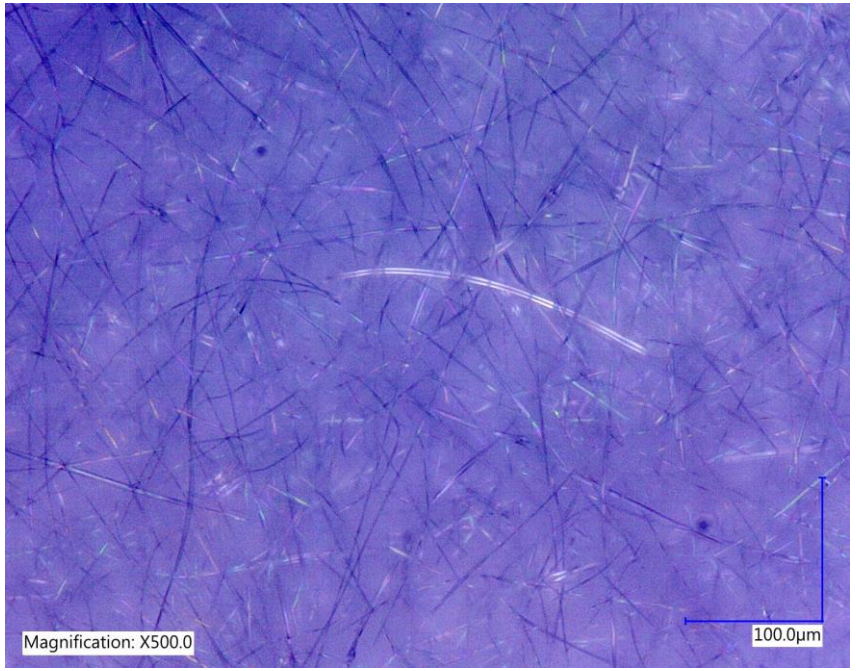




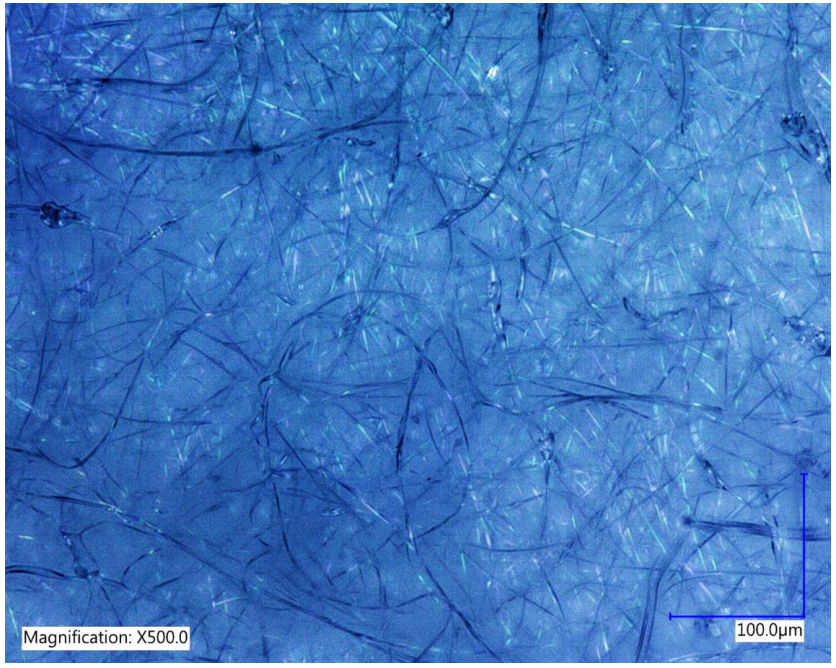
Microscopic examination of generated fiber of PVP polymer with different magnification

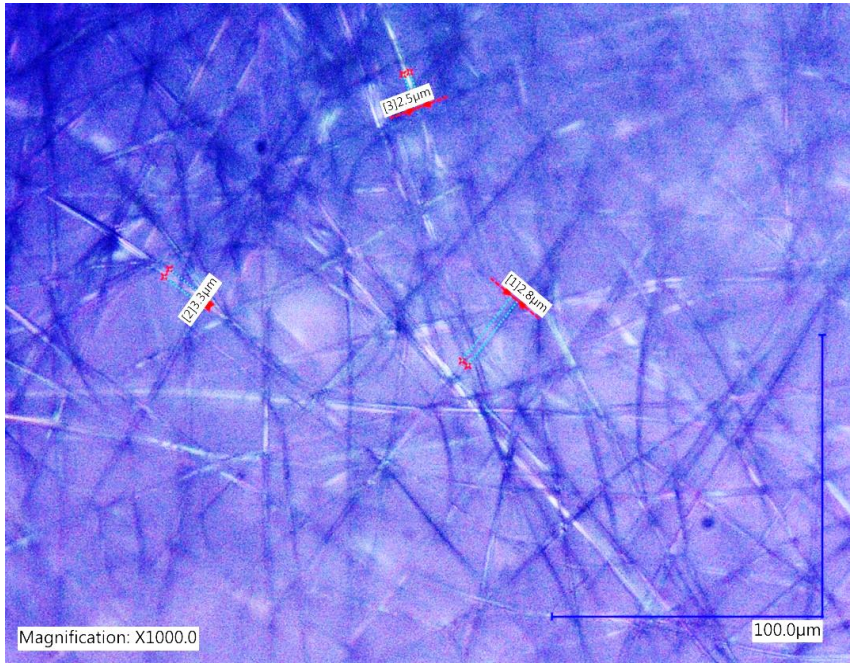


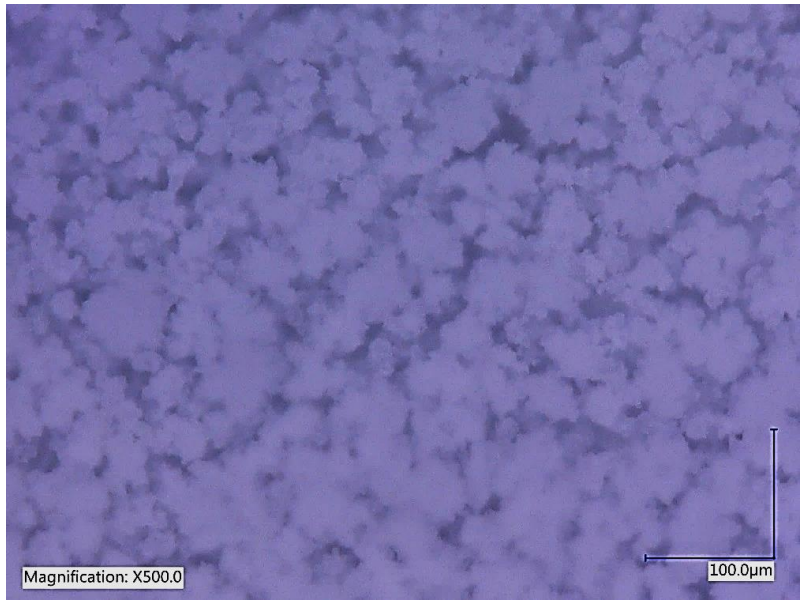


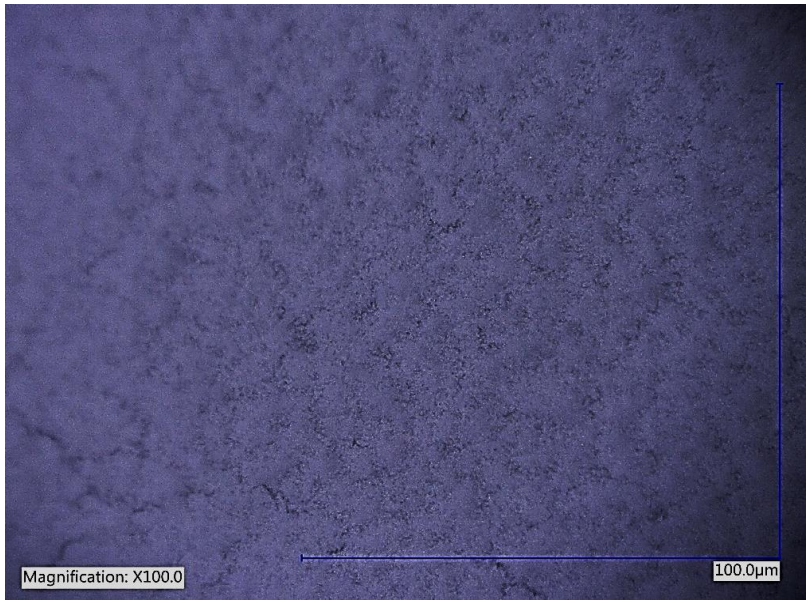


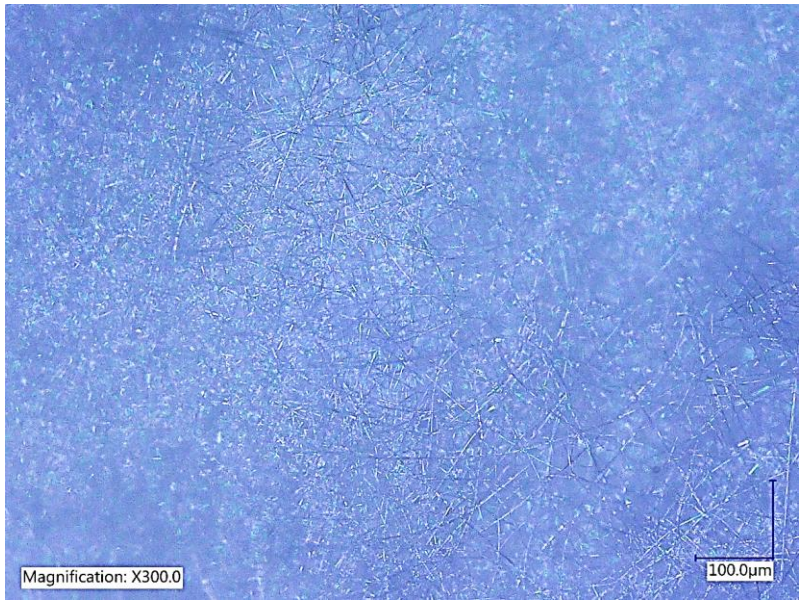
Microscopic examination of CA polymer fiber of different concentrations (5, 10, 11, 13, 15) % in acetone solvent

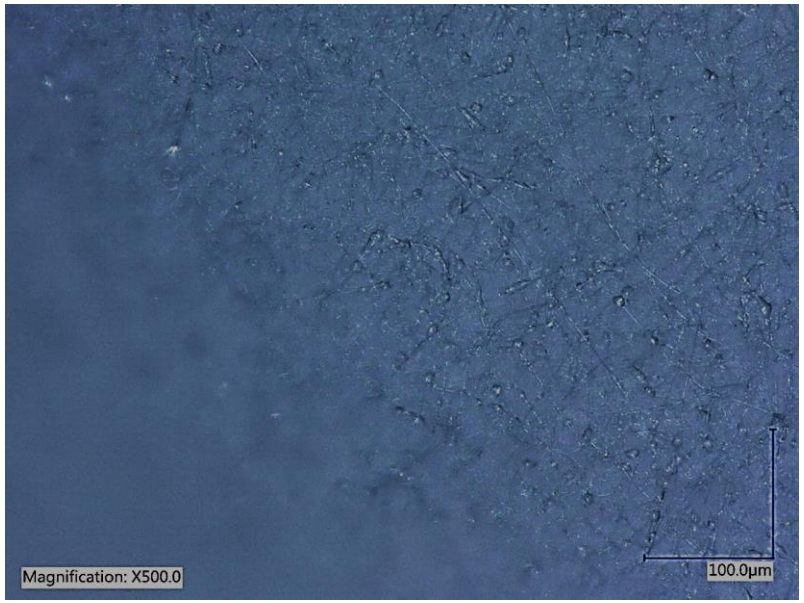


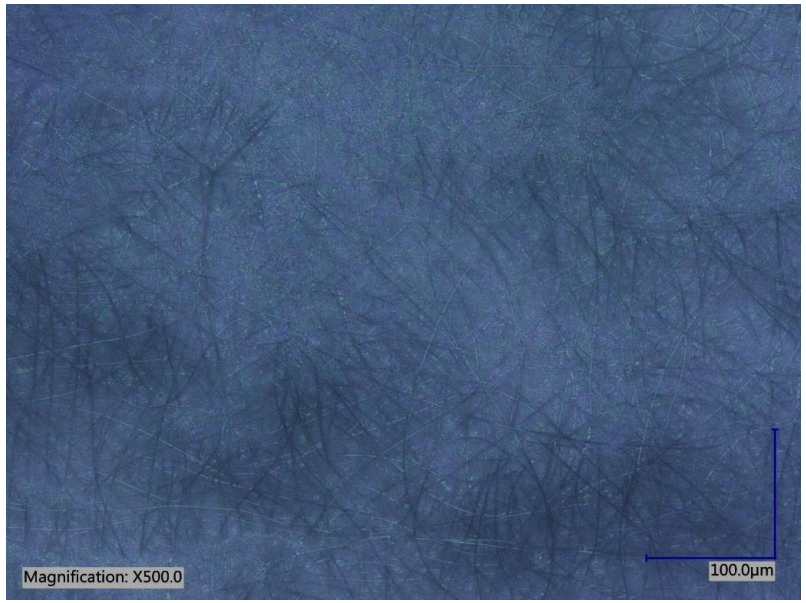


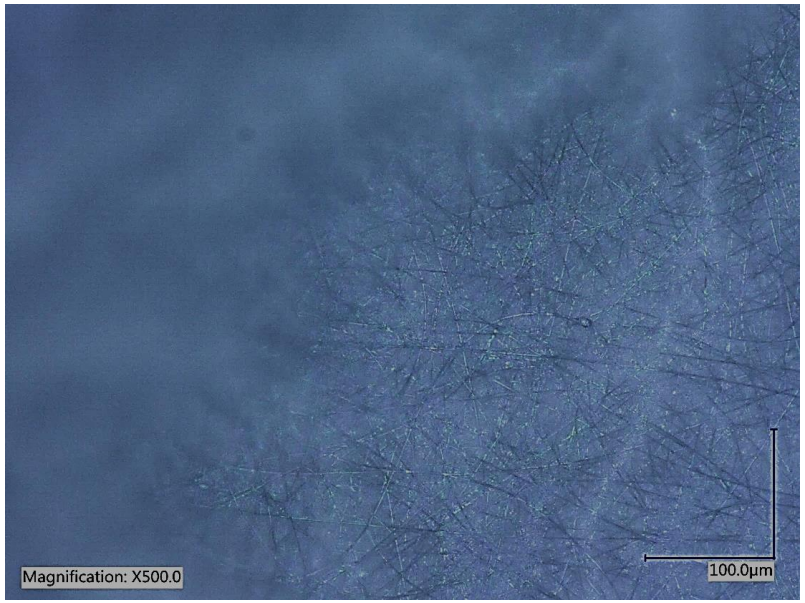




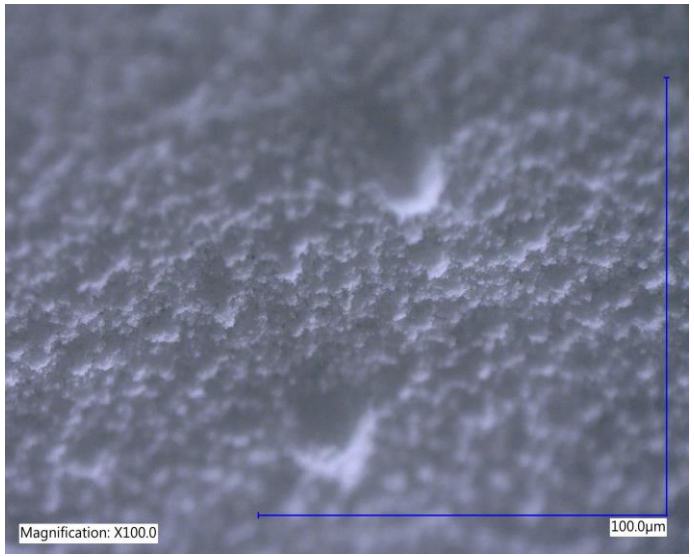


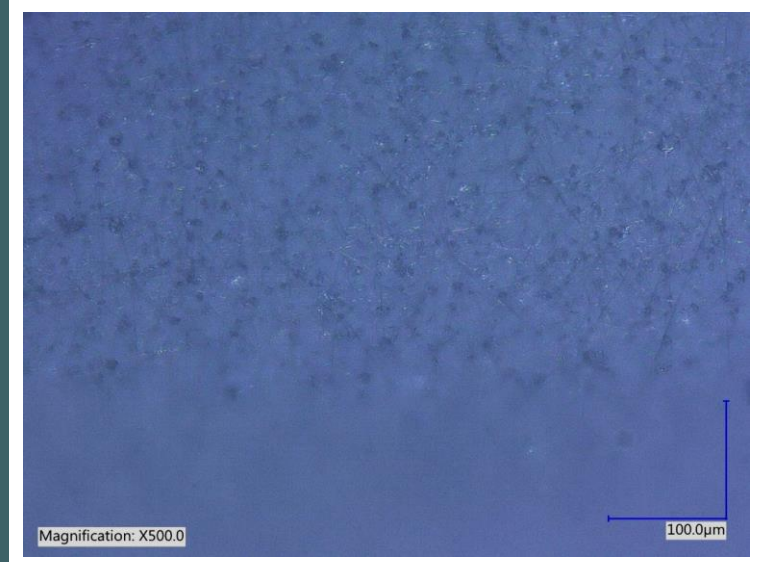


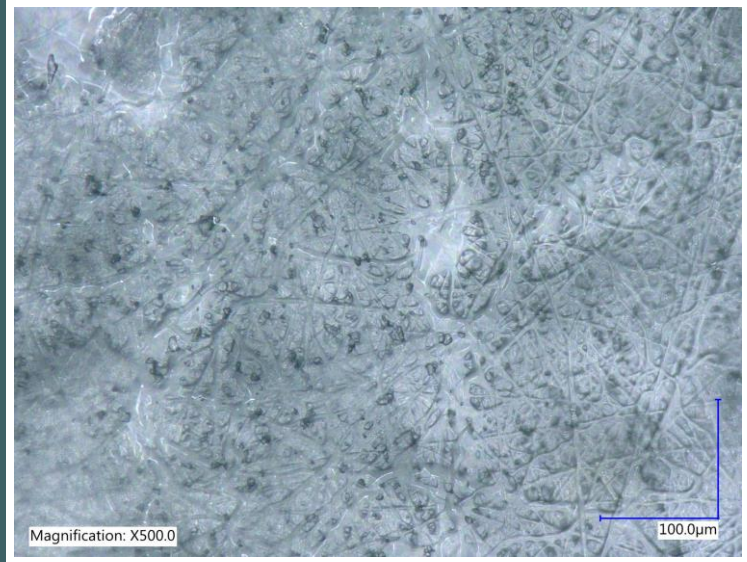


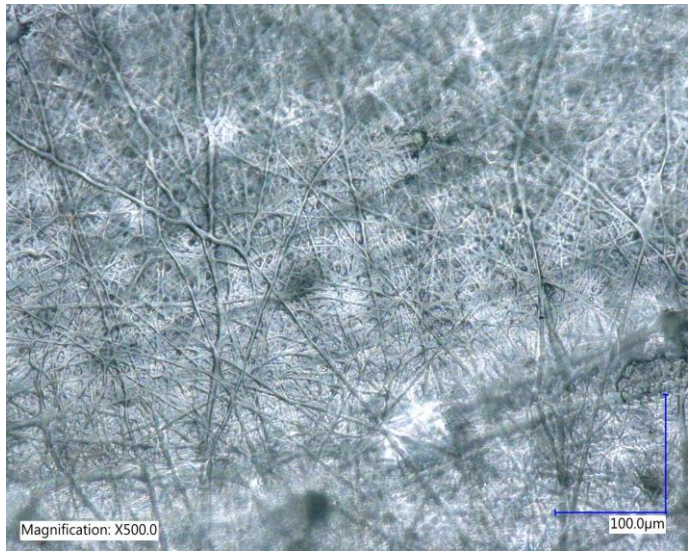


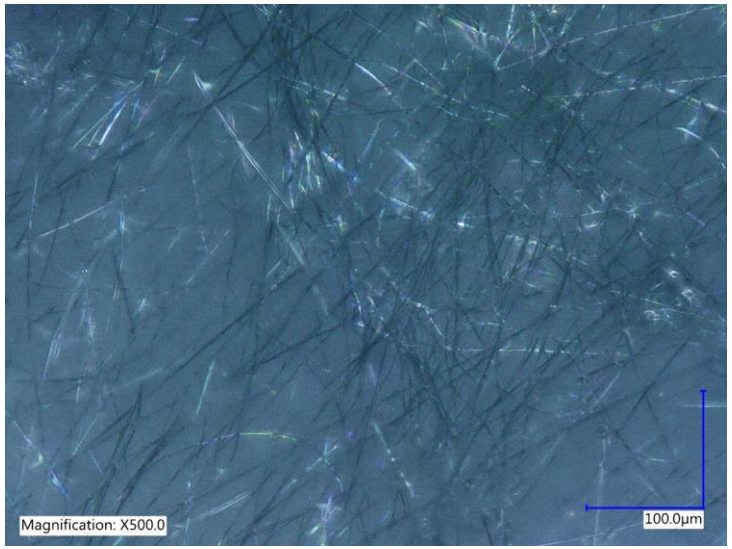
Microscopic examination of CA polymer fiber of different concentrations (5, 10, 15, 16, 18, 20) % in acetone–DMF (2:1)

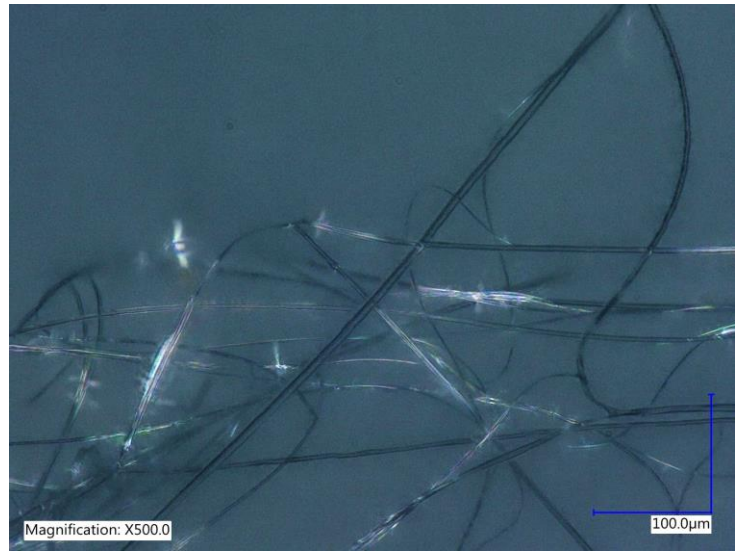








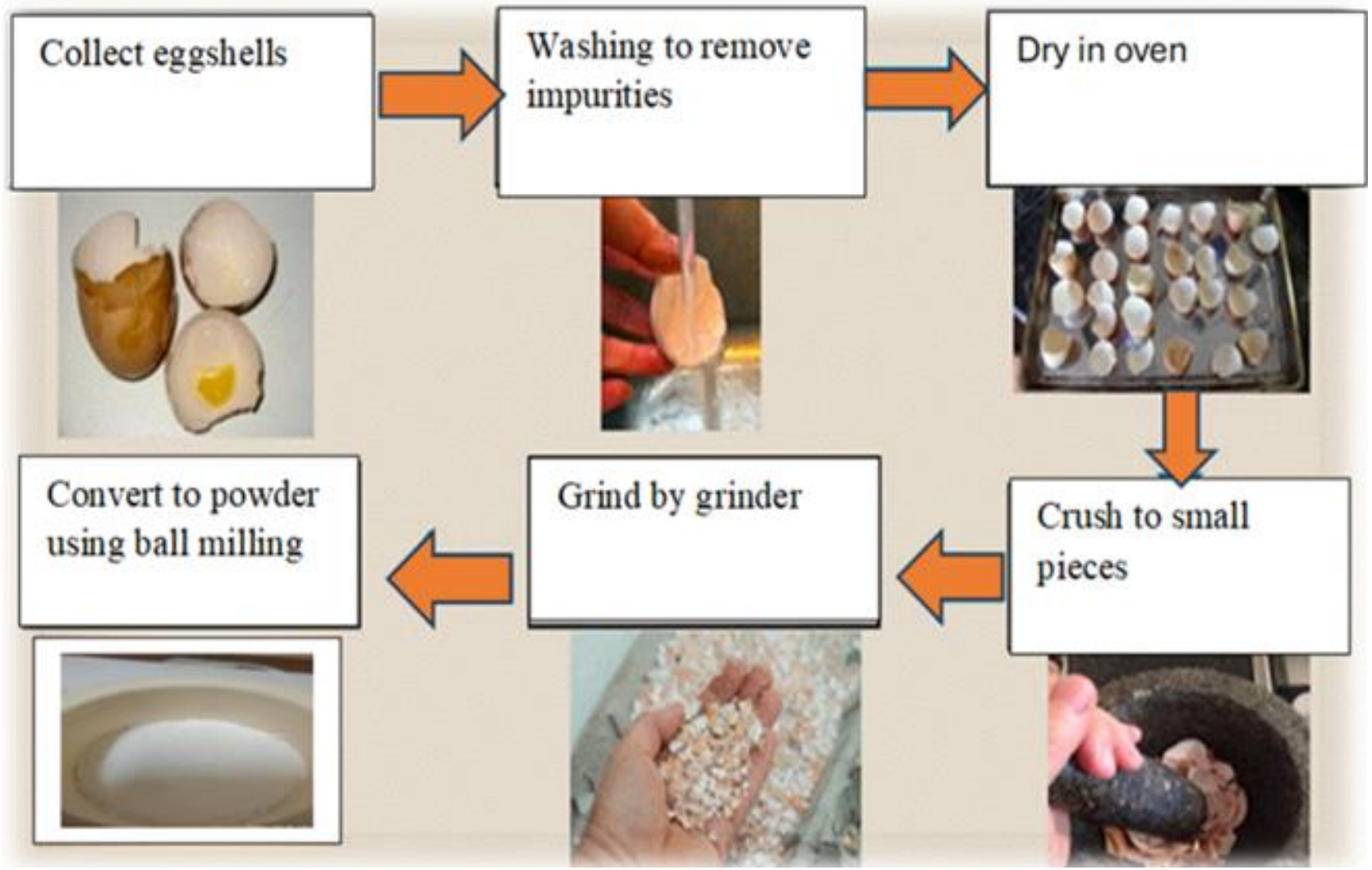




Microscopic examination of CA polymer fiber of different concentrations (5, 10, 11, 13, 15) % in acetone–acetic acid (2:1)

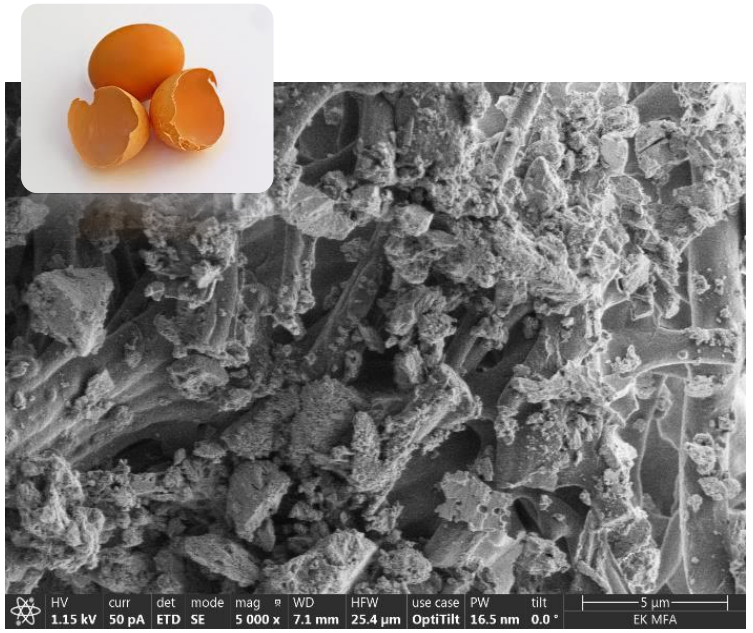
Eggshells as Bioactive Agent

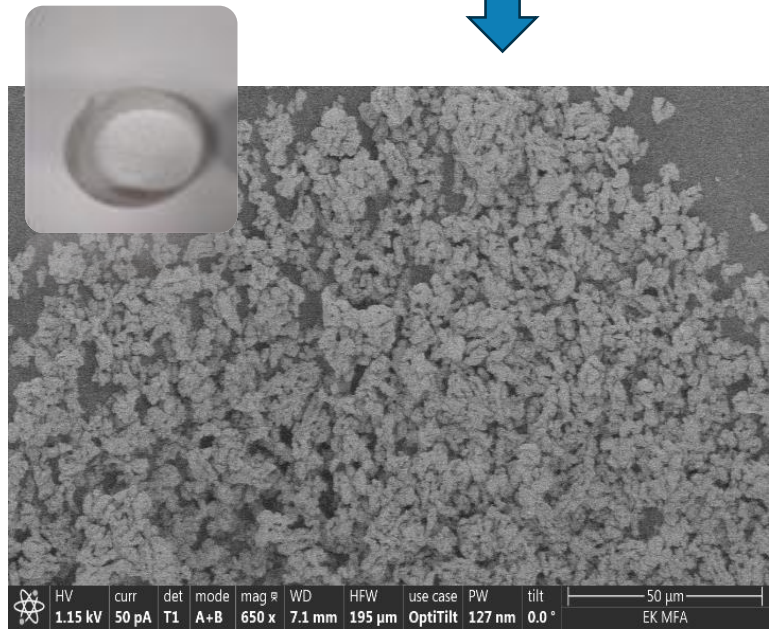
Fresh chicken eggs were collected and then the waste eggshell was initially washed and cleaned with tap water. Next, boiling water was used to remove any biological impurities stuck on the shells and dried in an electric oven. After that, the dried shells were broken by hand into small pieces before being ground to powder using ball mill machine.



Heat treatment

The eggshells have been subjected to heat treatment in the air at 900 °C for 12 hours, to extract CaO.



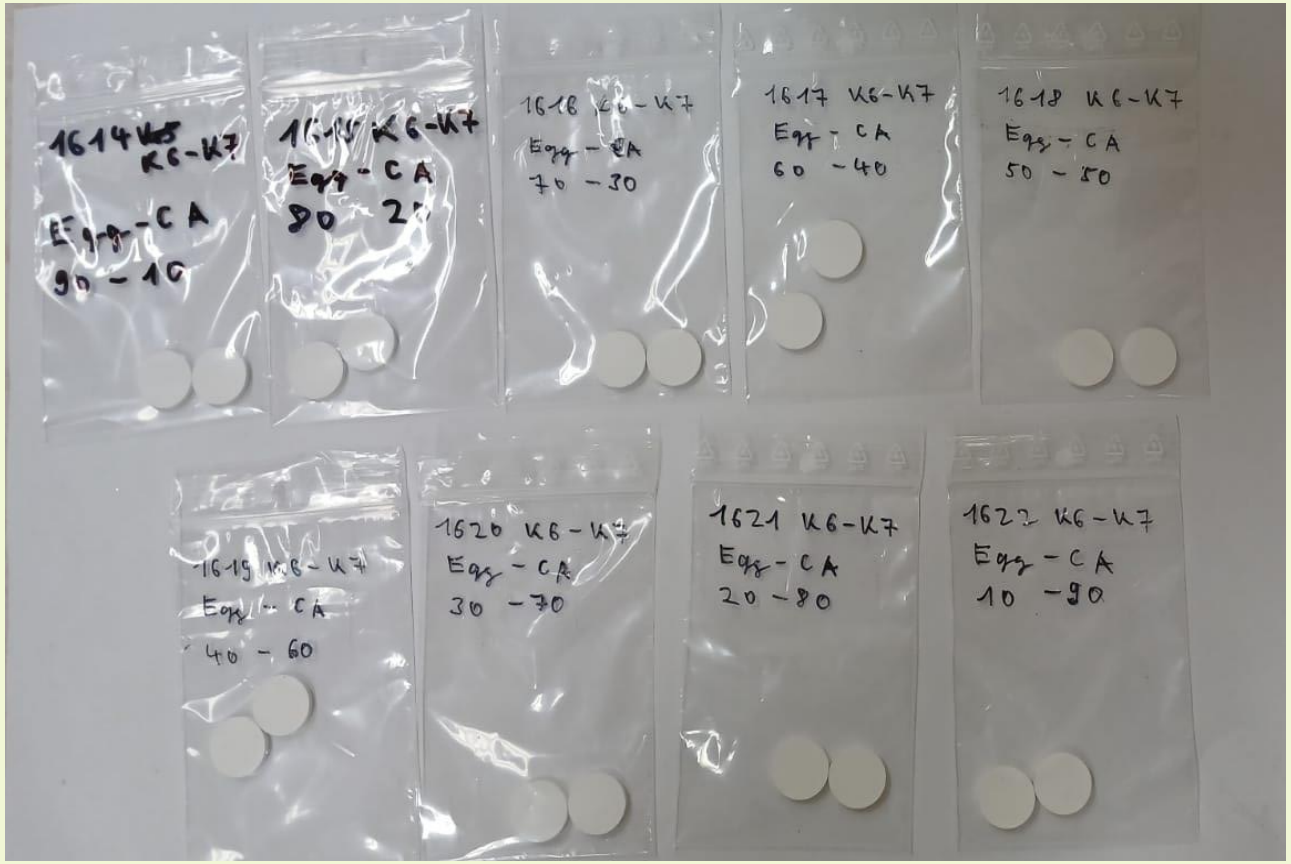


$$CaCO_3 \xrightarrow{\text{Calcination}} CaO + CO_2$$

Determine of mechanical integrity

Calcinated eggshell mixed with CA in different percentages (100-0) (90 -10) (80-20) (70 -30) (60 -40) (50-50) (40-60) (30-70) (20- 80) (10 -90) (0-100) using ball mall machine for a period of 3 hours to ensure good mixing between them.

Then the mixture of each ratio pressed under pressure to make a disc using a pressing machine with a pressure of 150 bar. These composites discs heat treated at different temperatures (180, 220) C^{o} to increase the compacting of the composite, the temperature used are under the melting point of CA to prevent decompose and burn of CA.

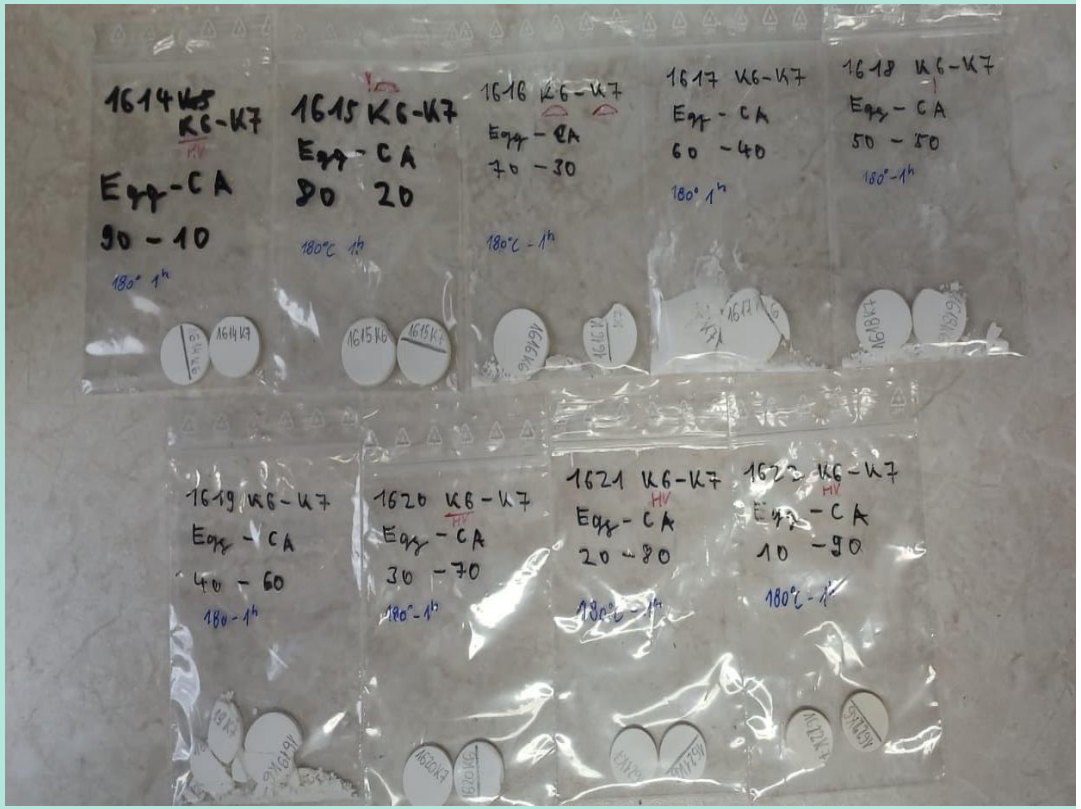


Determine of mechanical integrity

These composites discs heat treated at different temperatures (180, 220) C^{o} to increase the compacting of the composite, the temperature used are under the melting point of CA to prevent decompose and burn of CA.

Table (1) Composites after heat treatment 180 ^{o}C

CCaO-CA	Code	HV	Properties / appearance
(90 -10)	1614	0.6485	smooth without crack (samples became small)
(80-20)	1615	0.4675	smooth without crack (but samples bigger than 90-10
(70 -30)	1616	-------	Almost like 80-20
(60 -40)	1617	-------	One sample cracked and the second begins to crack
(50-50)	1618	-------	Smaller than 60-40 and almost smooth
(40-60)	1619	-------	Rough and begin to crack
(30-70)	1620	-------	Small and smooth
(20- 80)	1621	0.4103	Smooth no cracked but smaller
(10 -90)	1622	0.5357	Smooth no cracked and change color

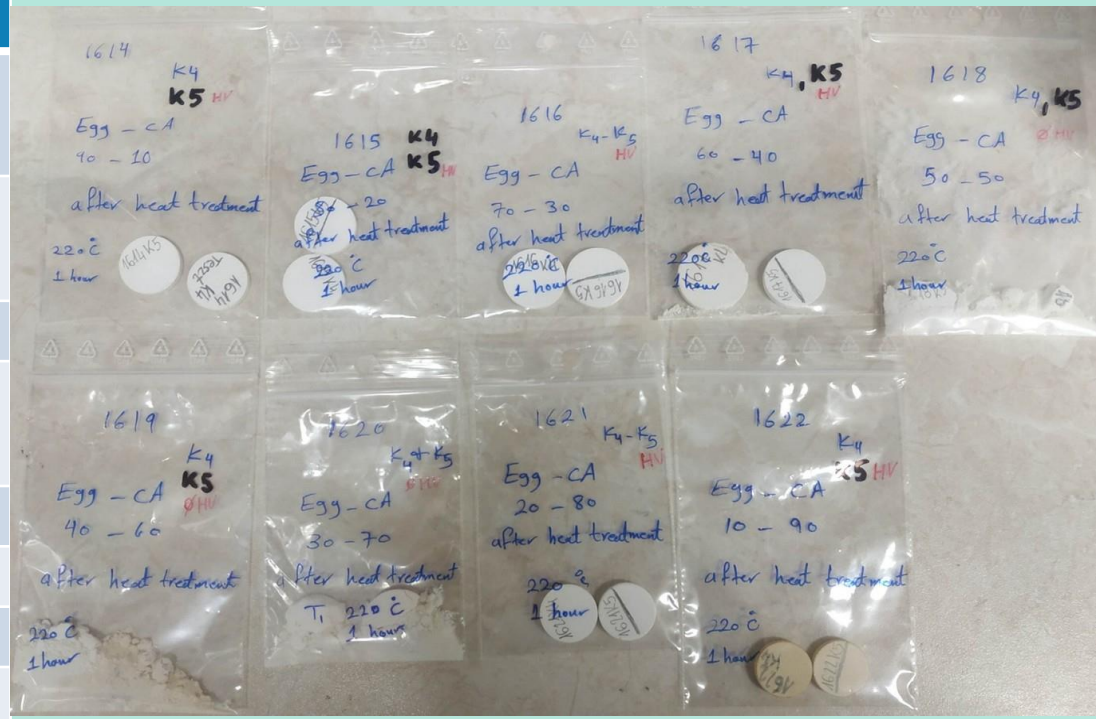


Determine of mechanical integrity

These composites discs heat treated at different temperatures (180, 220) C^{o} to increase the compacting of the composite, the temperature used are under the melting point of CA to prevent decompose and burn of CA.

Table (2) Composites after heat treatment 220 ^{o}C

CCaO-CA	Code	HV	Properties / appearance
(90 -10)	1614	0.4877	Soft and smooth without crack
(80-20)	1615	0.3167	Soft and smooth without crack
(70 -30)	1616		A little bit rough without crack
(60 -40)	1617		One sample rough and cracked another sample is just rough
(50-50)	1618		Cracked and turned to powder
(40-60)	1619		Cracked and turned to powder
(30-70)	1620		Turn to powder
(20- 80)	1621	0.314	Smooth no cracked but smaller
(10 -90)	1622	0.4116	Smooth no cracked and change color



Summery

01 Ethanol is proper solvent for production PVP polymer fiber, while acetone as single solvent and acetone/acetic acid as co solvent are a proper solvent for CA polymer fiber.

02 The combinations (90-10), (80-20), (20-80), and (10-90) are satisfactory, in both observations in table 1 and 2. Other are probably not good

03 The alternative blending ratios resulted in a powdered form.

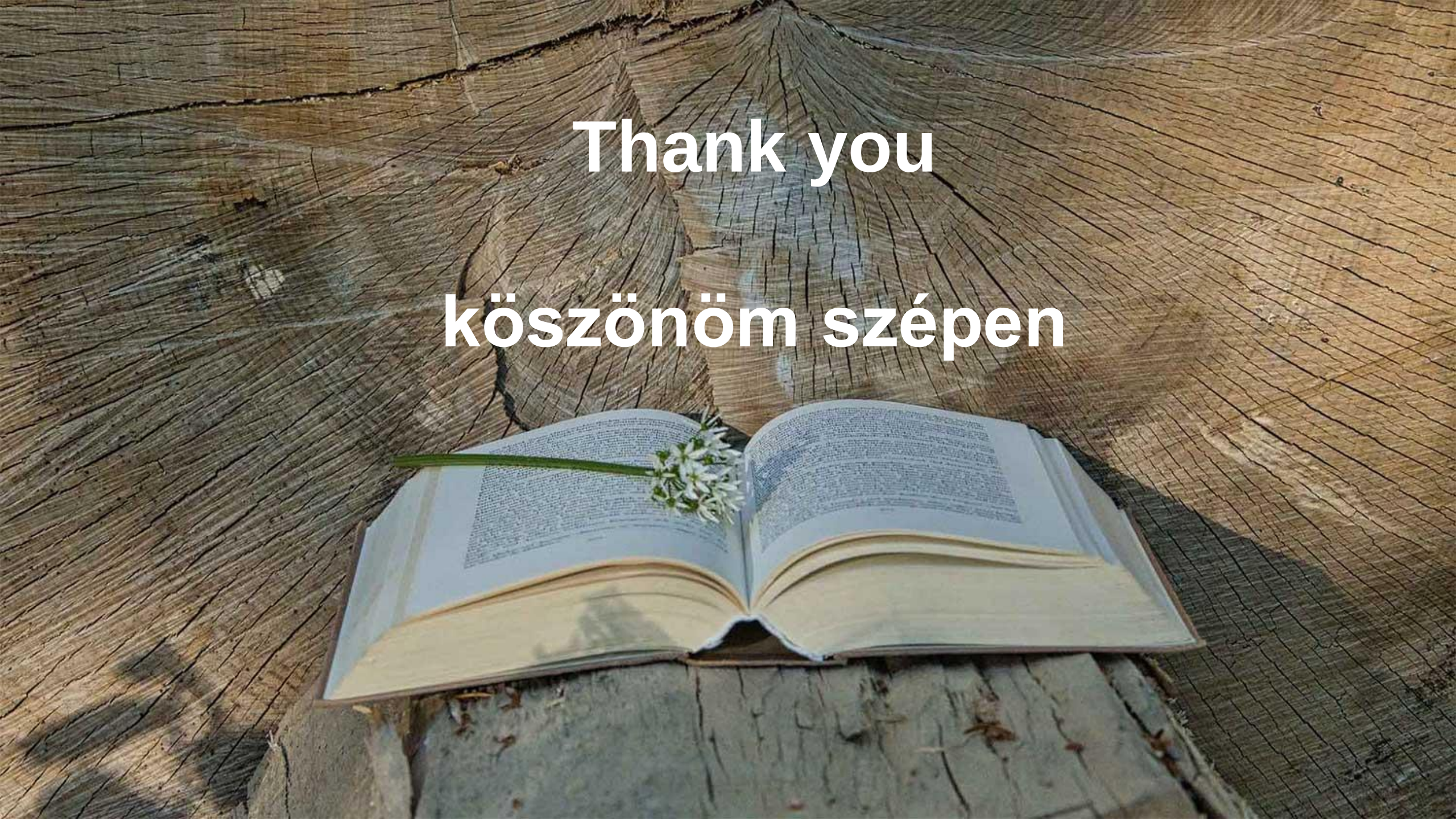
04 The concentration ratios (90-10), (80-20), (20-80), and (10-90) appear to function as both filler and matrix, making them suitable for production and compaction processes.

Courses and Credits:

Completed subjects from the beginning of the training programme	***Semester when the subject was completed***	***Credit***
Fundamentals of material science (Professor Maria Berkes).	***10/1/2024***	***6***
Biomaterials for medical applications (Professor Csaba Balázsi).	***10/1/2024***	***6***
Powder technology (Professor Csaba Balázsi).	***27/5/2024***	***6***
Transmission Electron microscopy (TEM) (Professor Katalin Balázsi).	***27/5/2024***	***6***
Selected chapters of material testing methods I: FTIR, (Professor Erzsébet Takács), SEM, STM, AFM (Professor Judit Telegdi)	***16/12/2024***	***6***
Chemical Cellulose (Professor Borsa Judit)	***6/1/2024***	***6***

Publication Papers:

No	*Title*	*Name of journal or conference*	*IF, Q*	*Total credit*	*%*	*Credit*
1	Hydroxyapatite-Based Natural Biopolymer Composite for Tissue Regeneration	Materials	5.8, Q2	36	100%	36
2	A Critical Review of Natural and Synthetic Polymer-Based Biological Apatite Composites for Bone Tissue Engineering	Composites Science	3.0, Q2	36	100%	36
3	Evaluation of Surface Roughness of 3D Printed Objects	3D Printing and Additive Manufacturing	2.3, Q2	36	100%	36
4	Effect of low-pressure plasma treatment on the thermal behavior of organo-modified montmorillonite nanoclay	Archives of Materials Science and Engineering	2.19, Q3	36	100%	36
5	The effects of black tea extracts on the corrosion inhibition of mild steel in acidic solution.	Nanomaterials Science & Engineering		24	100%	24
					Total	168


Thank you
köszönöm szépen