EXPERIMENT 2

Preparation of polyvinyl alcohol

The aim of this experiment is to get knowledge on method of continuous production of polyvinyl alcohol. At first step a polymerization of vinyl acetate occurs, and in subsequent step, polyvinyl acetate is converted into polyvinyl alcohol during alkaline alcoholysis. Polyvinyl alcohol has the same degree of polymerization as polyvinyl acetate, from which it was obtained. Scheme of apparatus for the preparation of polyvinyl alcohol is shown on Figure 1.

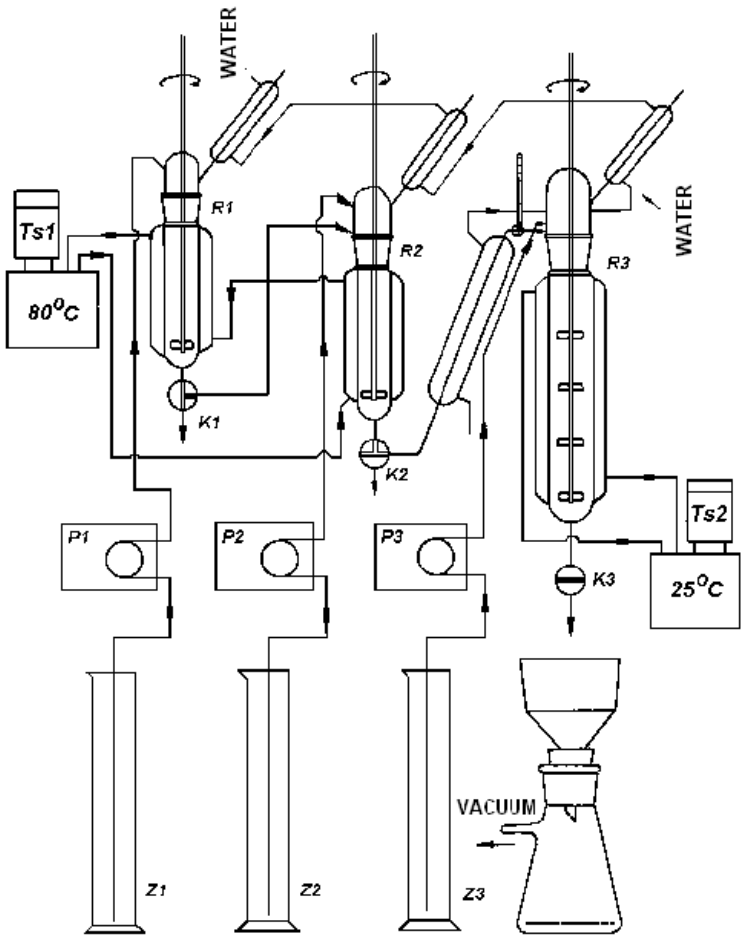


Fig. 1 Scheme of the installation (see the symbols explained in next)

To carry out experiment you have to sequentially perform the operations listed below. **The report from this laboratory shout include a resulting data sheet, worksheet (checklist) which will be marked with all the tasks, together with exact time when you started and finished any particular operation.**

1) In 500 mL conical flask prepare a solution of vinyl acetate, ethanol and benzoyl peroxide (quantities and concentrations will be given by instructor). Transfer the solution into graduated cylinder (Z1).
2) Turn on the stirrer and heating.
3) Open the flow of cooling water in both reactors
4) Open three-way tap (K1) from reactor 1 (R1) in position that allows flow of the solution to reactor 2 (R2).

5) Set three-way tap (K2) from reactor (R2) in a position preventing the flow of the solution to reactor 3 (R3)
6) Using a peristaltic pump (P1) transfer the solution to glass reactors (R1 & R2) equipped with a stirrers and reflux condensers.
7) Polymerization should be carried out under reflux for 1-2 hours. During the polymerization vigorous stirring must be ensured.
   NOTE: during the implementation of step 7, go forward with point 11 and then continue with the points 8-10.
8) Pour the methanolic NaOH solution into measuring cylinder (Z3), check the valve (K3) and turn on the stirrer in reactor 3 (R3) with peristaltic pump (P3). Transfer the NaOH solution into a reactor (R3).Regulate peristaltic pumps P1 and P2 till consumption of substrates will be the same in both cylinders. Start continuous production by writing down time, solution levels in cylinders Z1 and Z3.
9) Cut three filter paper sheets and weigh them with Petri dishes. Assemble a vacuum filtration kit and collect three fractions of polymer in continuous mode. Filtrate the fractions to separate polymer precipitate. Sign the names of persons performing the exercise, weigh and leave to dry. After drying, weigh again (next day).
10) Stop continuous production by switching off peristaltic pumps.
11) **Determination of acetate groups**. ***This part of experiment (analysis of polymer) is to be done simultaneously with point 7.***
    Determine the molecular concentration of NaOH by titration with 0.500M HCl (two checks titration with phenolphthalein as an indicator). Make four weighed portions of dried polyvinyl alcohol using analytical balance (about 200 mg with an accuracy of 1 mg), place them in four conical flasks, add 10 mL of hot distilled water and 10 mL of 0.5 M NaOH (pipette) to each flask. Heat to boil under reflux for 30 min. Turn off the heating and leave to cool down. Titrate the excess of NaOH 0.5N HCl with phenolphthalein as an indicator. Calculate the percentage of acetate groups:

$$\frac{}{}$$

where:
a - volume of 0.500 M solution of HCl used for titration of 0.5 M solution of NaOH
b - volume of 0.500 M solution of HCl used for titration of the sample,
0,0295 - conversion factor,
g - mass of analyzed sample of polymer.

12) **Reporting of the results.** The results should be written in worksheet. Calculate the efficiency of the process and the consumption of the monomer per mass unit of the product. Make a Sankey diagram.
13) Clean the glass and work space.

EXPERIMENT 2

Preparation of polyvinyl alcohol

Results sheet

Names of the students: ..... date.....
.....
.....

Raw materials:

solution S: vinyl acetate .....ml, alcohol .....ml, initiator .....g

hydrolyzing solution .....ml (contents: .....g of NaOH + ...ml of methanol)

Products:

Polyvinyl alcohol .....g, solution after the reaction .....ml

Time of initial reaction .....h. Duration of continuous process.....h

Flow rates:

Solution S .....ml/h, hydrolyzing solution.....ml/h

TITRATION RESULTS

Volume of HCl consumed in the titration of NaOH solution:

1. .....ml 2. .....ml 3. .....ml

average:……..

Titration analysis of weighed amounts of PVA:

Sample number	Amount of weighed PVA [g]	Volume of 0.5M HCl [ml]	Notes

Comments and notes:

Results accepted.....

date and signature of the instructor

The report of the exercise should include:

1. **Purpose of the exercise**

2. **Description of the experiment, including:**

a. general scheme of the apparatus (according to the rules posted on the website),

b. concise description of performed operations,

c. raw data (results sheet / data protocol signed by the instructor),

3. Results development:

a. chemical equations*,*

b. calculations,

c. Sankey diagram (according to the rules posted on the website)

d. Gantt chart (according to the rules listed below).

4. **Discussion of the results*.***

a. error sources,

b. yield.

5. **Conclusions. In particular:**

a. advantages and disadvantages of this method of polymerization*,*

b. economic side of the process (propose methods which enable to recover unused reagents),

c. toxicological aspects (see material safety data sheets),

d. influence of the way of industrial process on environment, suggestions for improvement,

e. comment on whether the aims of the experiment have been achieved.

TABLE OF OPERATIONS FOR THE EXPERIMENT **Nr. 2**

student A: …… student B: …… student C: ……

	Steps to perform	**Performing student**	**Time**	
			Start	**End**
1	Reading the Material Safety Data Sheet (MSDS)	all		
2	Measure vinyl acetate and ethanol.	A		
3	Weigh and dissolve the initiator.	B		
4	Turn on the thermostat.	C		
5	Check the seals.	C		
6	Open the cooling water valve.	C		
7	Transfer the solution into a reactor.	all		
8	Starting (initialization) of the process. **(1-2 hours. At this time, please perform steps 18-25)**	all		
9	Check the valve and turn on the stirrer in reactor 3.	A		
10	Transfer the NaOH solution into a reactor 3.	A		
11	Start continuous production (time and amount of liquids have to be written in lab-book!).	A,B		
12	Cut three filter paper sheets and weigh them with Petri dishes.	B		
13	Assemble a filtration kit.	A		
14	Collect three fractions of polymer in continuous mode.	A		
15	End continuous production. (time and amount of liquids have to be written in lab-book!).	A		
16	Filtrate the fractions.	B		
17	Dry the polymer.	A		
18	Turn on the automatic burette.	C		
19	Set the titre of NaOH on 0.500 M hydrochloric acid.	C		
20	Weigh four samples of the polymer into conical flasks.	B		
21	Hydrolyze the polyvinyl acetate samples under reflux condenser.	B		
22	Cool and titrate the samples.	C		
23	Turn the heating off.	C		
24	Turn off automatic burette.	C		
25	Calculate the amount of acetate groups.	all		
26	Clean the glass.	all		
27	Turn the thermostat off.	C		
28	Turn off the cooling water.	C		
29	Check the amount of glass and equipment.	all		
30	Fill the data protocol to be signed by instructor.	all		
31	Check and clean analytical balances.	all		

Report of the experiment 2 (template) date.....

Preparation of polyvinyl alcohol

Names of the students:

....

1.Purpose of the experiment:

2. The sequence and description of activities performed:
(attach checklist)

3. Results calculation:
Weight of obtained polymer:.....g

Material balance for one hour of continuous mode

Substance	In [g/h] [1]	Out [g/h] [2]
ethanol		
initiator		
monomer (vinyl acetate)		
methanol		
NaOH		
polymer		
after reaction mixture		
	∑=	∑=

Yield calculation for 1 hour at continuous mode:

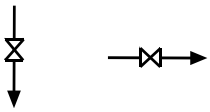
Calculation the monomer consumption per unit of product mass:

Calculation of the amount of acetate groups:

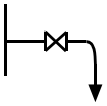
[1] The materials entering the process.
[2] The materials leaving the process.

Discussion of results and conclusions (according to instructions, on pages attached separately).

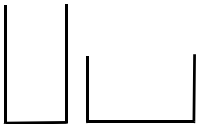
SYMBOLS USED FOR THE IMPLEMENTATION OF APPARATUS SCHEME



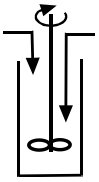
valves



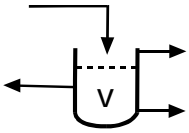
tap



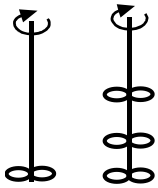
open container (horizontal and vertical)



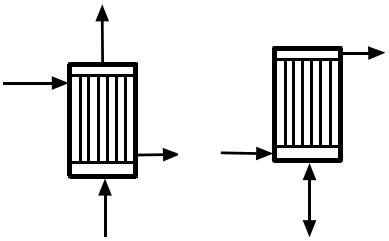
Precipitation apparatus



Filtration kit

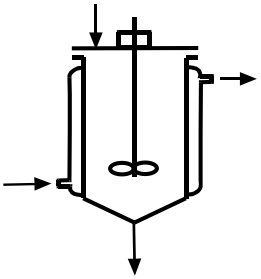


stirrers

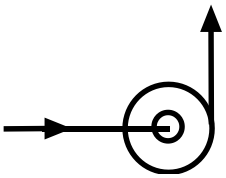


heat exchanger

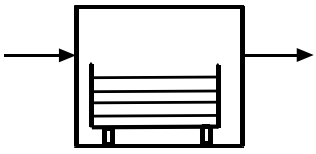
reflux condenser



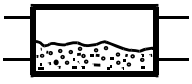
reactor with heating mantle and stirrer



pump



dryer chamber



dryer

How to prepare the Gantt's chart

The Gantt's Chart is a two-dimensional diagram applied for a visual description of the project.

From correctly-made chart we can quickly read the structure of the project, the tasks (activities) and the time of their performance (start and end time, and the order of tasks performed). Gantt Chart can be made by hand (on graph paper) or by the use of computer. It is always necessary to unambiguously determine the time intervals. To prepare Ganntt's Chart you have to use the checklist sheet on which you noticed the start time and end time of each activity.

activity name	start	end
A	8:30	8:45
B	8:35	8:45
…	….	….
…	….	….
N	12:25	12:50
O	12:50	13:00

On the axis of ordinates, please put the name of the activity and on the axis of abscissae put time scale (eg hours with division into minutes, quarters, etc.):