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Research article

DETERMINATION OF THE OPTIMAL PARAMETERS FOR MULTIPLE USING OF BAROMEMBRANES FOR PURIFICATION OF GEORGIAN POPULAR WINES

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Abstract

The fluorine-organic baromembranes for purification of Georgian popular wine have been used. The corresponding apparatus is constructed. Optimal parameters of filtration and regeneration of the used baromembranes have been established.

Key words: baromembrane, filtration, Georgian wines, optimal, parameter, regeneration.

Introduction

Membrane technology occupies a special place in liquid food production in respect of filtration, fractioning, concentration and microbiological stabilization [1-4].

The most of liquid foodstuffs are multicomponent systems. They contain suspended, colloid, insoluble substances which also have complicated content. Correspondingly filtration of concrete liquid products requires determining of filtration technological parameters correctly match to the regenerants which provide complete regeneration and improve regeneration technology modes that are the main object of our research. By using baromembrane [5] the process of filtration may become cheaper, technologically simpler and cause increase of quality indexes [6-18]. In addition, it can use multiply [2].

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Results and Discussion

We have researched Georgian popular wines –”Tsolikauri“,”Tsitska“, Rkatsiteli”, ”Chkhaveri“, ”Saperavi “. These wines, especially ”Saperavi“, along with fine taste have also good antioxidant properties [18]. The results of carried out researches is given in tables 1-10.

Table 1. Chemical Content of wine ”Tsolikauri“

Gathering place of grapes (wines made in families)	Specific weight	Alcohol content, vol. %	Sugar content, %	Mass concentration, g/L				
				Titrating acidity of tartaric acid equivalent	Volatile acid of acetic acid equivalent	Extract	Tanning agents	Sol
Sviri village, Zestafoni region	0.9913	12.5	0.08	7.5	0.95	16.6	0.26	2.01
Baghdadi region	0.9935	13.9	0.32	9.3	0.66	20.0	0.35	1.96
Khvantchkara village, Ambrolauri region	0.9926	12.8	0.12	10.1	1.54	23.5	0.52	2.08
Terjola village, Terjola region	0.9916	11.4	0.08	6.9	0.66	20.1	0.18	2.05
Keda village, Keda region, Adjara	1.0029	12.6	2.62	6.9	0.75	31.8	0.58	2.03

Table 2. Chemical content of wine ”Tsitska“

Gathering place of grapes	Specific weight	Alcohol content, vol. %	Sugar content, %	Mass concentration, g/L						
				Titrating acidity of tartaric acid equivalent	Volatile acid of acetic acid equivalent	Extract	Tartaric acid	Tanning agents	Sol	Glyce-rine
Sviri,, Zestafoni region	0.9906	14.2	2.0	6.6	0.450	20.9	2.40	0.41	-	6.8
Terjola,	0.9913	12.3	0.2	5.1	0.64	18.8	2.30	0.28	1.83	7.8
Ghviara, Ambrolauri region	0.9918	13.3	0.1	7.8	0.71	23.0	3.23	0.60	-	-

Table 3. Chemical content of wine”Rkatsiteli“

Gathering place of grapes	Specific weight	Alcohol content, vol. %	Sugar content, %	pH	Mass concentration, g/L						
					Titrating acidity of tartaric acid equivalent	Volatile acid of acetic acid equivalent	Extract	Tartaric acid	Tanning agents	Sol	Glyce-rine

Akhmeta	0.9939	10.2	-	3.66	4.2	0.83	20.4	1.62	1.70	2.66	5.5
Tsinandali	0.9936	19.9	-	3.66	5.0	1.01	20.7	2.09	1.89	2.09	5.3
Gurjaani	0.9932	11.4	-	3.47	4.9	0.95	21.6	1.90	2.24	2.00	5.6
Kvareli	0.9941	10.6	-	3.45	5.6	0.97	20.8	1.92	1.76	2.1	5.9
Telavi	0.9941	11.4	-	-	7.1	0.70	25.8	-	0.39	1.40	68

Table 4. Chemical content and organoleptic properties of wine”hkhaveri“

Gathering place of grapes	Specific weight	Alcohol content, vol. %	Sugar content, %	Mass concentration, g/L							Organoleptic properties
				Titrating acidity of tartaric acid equivalent	Volatile acid of acetic acid equivalent	Extract	Tartaric acid	Tanning agents	Sol	Glyce-rine	
Bakhvi village, Makharadze region	0.9934	11.2	0.13	8.1	0.80	2.5	22.6	0.41	2.1	5.5	amber yellow, pink, pleasant, enough high acidity, aromatic taste
Dabla Tsikhe, Chokhatquri region	0.9935	10.0	0.80	7.8	0.94	3.24	17.0	0.59	2.5 6	5.3	light straw coloration, light pleasant taste
Keda village, Keda region	0.9901	9.3	0.145	7.9	1.10	2.98	-	0.52	-	-	Straw coloration, fruit aroma, high, but acceptable acidity, light taste

Table 5. Content and properties of Georgian white wines

	Wine production method		
	Kakhetian (Rkatsiteli)	Imeretian (Tsolikauri)	European (”Rkatsiteli“)
Chemical content			
Extract, g/dm^3	24.7	21.2	19.1
General			
Ethanol, vol.%	11.28	11.46	11.52
Glycerine, g/dm^3	12.68	9.76	9.48
Sugar content, %	8.8	19.1	19.2
Wine sugar content,%	0.34	0.1	0.1
Monosaccharide ,g/dm^3			
Pentoses	177.5	157.5	170.0
Hexoses	127.5	145.0	187.5
Organic acids, g/dm^3			
Tartaric acid	2.59	3.48	2.61
Citric acid	0.59	0.69	0.70
Malic acid	1.32	1.53	1.87
Lactic acid	0.93	0.88	0.53
Phenols, mg/dm^3	1200.0	620.0	306.0
Catechines, mg/dm^3	250.0	35.0	30.0
Leukoanthocyanins, mg/dm^3	600.0	176.0	232.0
Mineral content, mg/dm^3	1350.00	580.00	1080.00
K	14.00	11.00	1.40
Na	125.00	63.00	97.00
Ca	25.50	18.50	26.00
Fe	no	no	no

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Cd, Co, Pb	0.62	0,40	100.00
Ni	3.60	2.21	2.72
Zn	0.02	0.02	0.01
Li	130.00	110.00	100.00
Mg	1.50	5.70	2.10
Mn	0.50	0.30	0.50
Sr	0.30	0.20	0.30
Cr	79.10	traces	64.80
Aromatic substances,	traces	traces	-
mg/dm^3	0.89	1.03	1.006
acetaldehyde	17,70	61,2	43.90
ethyl formate	350.70	33.07	106,10
acetone (standard)	9.30	10.5	10.00
ethyl acetate	103.80	32.97	147.26
ethanol	38.10	66.25	87.30
methanol	3.37	8.29	1.60
propanol	traces	0.90	0.80
esobutanol	18.6	24.46	19.85
n-butanol	89.39	207.58	93.80
ethylcapronate	0.62	0.87	0.86
methyl-2-butanol-1	traces	1.35	-
methyl-3-butanol-1	traces	-	-
n- amyl alcohol (standard)	0.80	-	-
unidentified substance	1.37	3.15	traces
unidentified substance	0.37	traces	-
ethyl enanthate	5.25	22.05	9.90
hexanol	3.80	1.90	1.60
unidentified substance	3.30	2.70	0.95
ethyl lactate	3,50	5.04	7.28
ethyl caprylate	traces	traces	-
unidentified substance	traces	-	-
furfural	106.30	81.9	92.58
unidentified substance	0.97	traces	0.14
unidentified substance	44,06	36.75	50.83
2,3-butilene	6.50	traces	1.5
ethyl caprinate	0.56	traces	0.87
glycol	6.00	1.2	1.27
unidentified substance	4.00	traces	1.09
unidentified substance	1.40	traces	0.37
unidentified substance	-	-	-
unidentified substance			
unidentified substance	1.485	0.530	0.98
unidentified substance			
	43.90	40.80	50.60
Physical properties of wine	58.80	41.20	60.60
optical density at 275 nm			
buffer capacity, mg.eq./dm^3			
as per acid			
as per alkali			

The samples for wine analysis shown in the tables were taken one year after wines producing. All wines contain lots of suspended substances of compound content. That is why, for membrane producing we chose thermo- and chemically stable polymer – polytetrafluoroethylene. We processed the plate containing secondary polytetrafluoroethylene and produced asymmetric membrane. The average porosity of produced membrane were determined by means of preliminary researches and theoretical calculations (average size of pores 100-120 nm).

We produced the filter-press type microfiltration assembly to provide its easy disassembling and washing with reverse flow (Fig. 1).

Figure. 1. Filter-press type microfiltration experimental apparatus

Based on experimental data, we show the variant of hydraulic scheme, which is shown on Fig 2.

Proceeding from 10 various wine filtration results, we present the results of 3 various wines filtration, because the rest wines filtration results are similar to these 3 kinds of wine. Namely, in Keda winery ("Kakhetian Traditional Wine" – KTW), we provided filtration of wine produced from Rkatsiteli grapes. Analysis of filtered wine proved correspondence to final product state and it was bottled for sales purpose. The results of wine filtration are shown in Table 6.

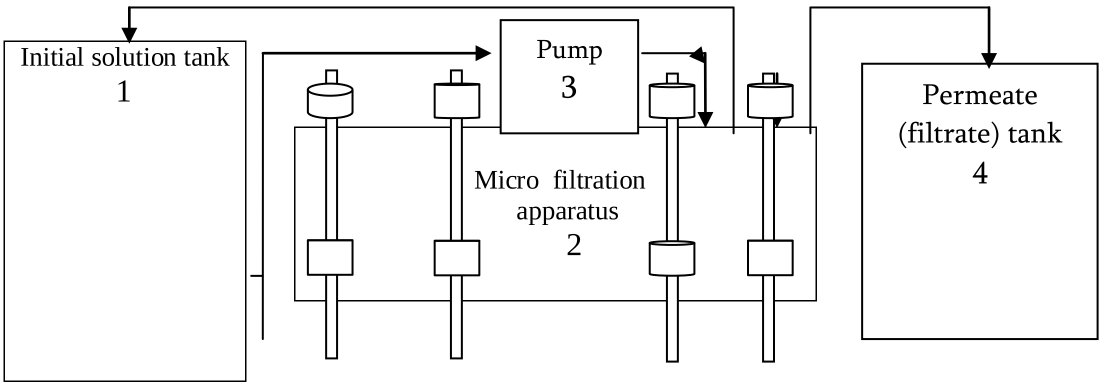

Figure 2. Hydraulic scheme of microfiltration experimental apparatus
1. Initial solution tank; 2. Microfiltration apparatus; 3. Pump; 4. Permeate (filtrate) tank

Table 6. Filtration results of white wine "Rkatsiteli"

№	Working pressure, P atm.	Quantity of filtered wine, l	Filtrate q-ty, l/sec.	Apparatus capacity, $l/m^2 \cdot h$	Remark
1	0.8	initial	5/76	475	wine
2	0.8	100	5/78	460	temperature
3	0.8	200	5/80	450	2^0C
4	0.8	300	5/82	440	

5	0.8	400	5/84	420	
6	0.8	500	5/88	410	
7	0.8	600	5/90	400	
8	0.9	700	5/94	385	
9	0.9	800	5/96	380	
10	0.9	900	5/100	360	
11	0.9	1000	5/104	345	
12	0.9	1100	5/108	333	
13	0.9	1200	5/112	320	
14	1.0	1300	5/120	300	
15	1.0	1400	5/124	290	
16	1.0	1500	5/130	280	
17	1.0	1600	5/136	260	
18	1.0	1700	5/142	250	
19	1.0	1800	5/150	238	
20	1.0	1900	5/160	225	
21	1.0	2000	5/170	213	
22	1.0	2100	5/180	200	
23	1.0	2200	5/190	188	
24	1.0	2300	5/200	180	
25	1.0	2400	5/214	168	
26	1.0	2500	5/230	155	
27	1.0	2600	5/250	142	
28	1.0	2700	5/280	127	
29	1.0	2800	5/320	113	
30	1.0	2900	5/360	100	
31	1.1	3000	5/400	90	
32	1.1	3100	5/440	83	
33	1.1	3200	5/480	75	
34	1.2	3300	5/520	68	
35	1.2	3400	5/570	63	
36	1.2	3500	5/620	58	
37	1.3	3600	5/680	53	
38	1.3	3700	5/740	48	
39	1.3	3800	5/800	45	
40	1.3	3900	5/870	40	
41	1.4	4000	5/940	38	
42	1.4	4100	5/1020	35	
43	1.4	4200	5/1100	33	
44	1.4	4300	5/1190	30	
45	1.4	4400	5/1280	28	
46	1.5	4500	5/1400	25	
47	1.5	4600	5/1540	23	
48	1.5	4700	5/1700	21	
49	1.5	4800	5/1880	19	
50	1.5	4900	5/2100	17	
51	1.5	5000	5/2400	15	
52	1.5	5100	5/2800	14	
53	1.5	5200	5/3200	12	
54	1.5	5300	5/3600	10	

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In order to visualize the changes of microfiltration assembly capacity we present the graphical results (Fig. 3).

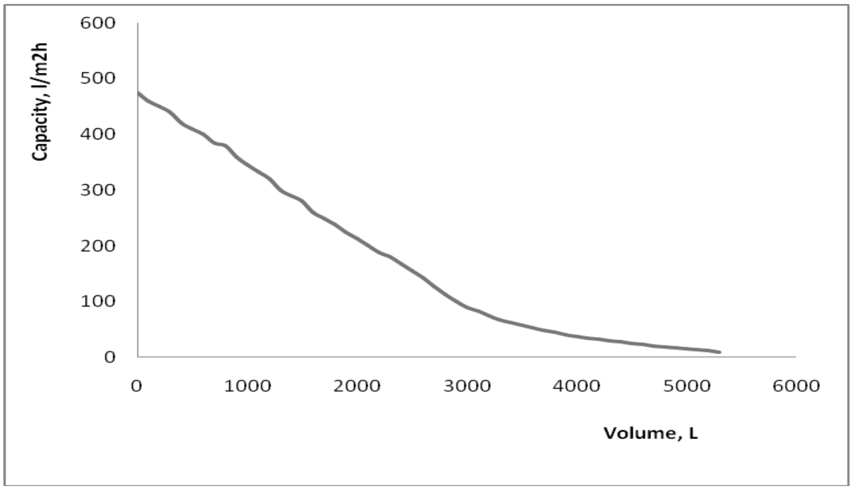

Figure 3. Relation of the apparatus capacity and quantity of filtered wine during filtration of white wine”Rkatsiteli“

After restoration of apparatus capacity we performed filtration of 500 L of wine produced from “Dzvelshavi” grapes from the family of well known Tskaltubo region. Microfiltration apparatus purified the wine. As a result of were shown that the filtrate was clean from suspended substances, besides quantities of all the compounds dissolved in the wine were the same as in the initial wine (before filtration). The results of changes of apparatus capacity are given in Table 7.

Table 7. The results of filtration of red wine”Dvelshavi“

№	Working pressure, P atm.	Q-ty of filtered wine, l	Filtrate q-ty, l/sec.	Apparatus capacity, $l/m^{2}\cdot h$	Remark
1	0.8	initial	5/76	475	wine
2	0.8	100	5/78	460	temperature
3	0.8	200	5/80	450	2^{0}C
4	0.8	300	5/82	440	
5	0.8	400	5/84	420	
6	0.8	500	5/88	410	
7	0.8	600	5/90	400	
8	0.9	700	5/94	385	
9	0.9	800	5/96	380	
10	0.9	900	5/100	360	
11	0.9	1000	5/104	345	
12	0.9	1100	5/108	333	
13	0.9	1200	5/112	320	
14	1.0	1300	5/120	300	
15	1.0	1400	5/124	290	
16	1.0	1500	5/130	280	
17	1.0	1600	5/136	260	
18	1.0	1700	5/142	250	
19	1.0	1800	5/150	238	
20	1.0	1900	5/160	225	
21	1.0	2000	5/170	213	
22	1.0	2100	5/180	200	
23	1.0	2200	5/190	188	

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24	1.0	2300	5/200	180	
25	1.0	2400	5/214	168	
26	1.0	2500	5/230	155	
27	1.0	2600	5/250	142	
28	1.0	2700	5/280	127	
29	1.0	2800	5/320	113	
30	1.0	2900	5/360	100	
31	1.1	3000	5/400	90	
32	1.1	3100	5/440	83	
33	1.1	3200	5/480	75	
34	1.2	3300	5/520	68	
35	1.2	3400	5/570	63	
36	1.2	3500	5/620	58	
37	1.3	3600	5/680	53	
38	1.3	3700	5/740	48	
39	1.3	3800	5/800	45	
40	1.3	3900	5/870	40	
41	1.4	4000	5/940	38	
42	1.4	4100	5/1020	35	
43	1.4	4200	5/1100	33	
44	1.4	4300	5/1190	30	
45	1.4	4400	5/1280	28	
46	1.5	4500	5/1400	25	
47	1.5	4600	5/1540	23	
48	1.5	4700	5/1700	21	
49	1.5	4800	5/1880	19	
50	1.5	4900	5/2100	17	
51	1.5	5000	5/2400	15	
52	1.5	5100	5/2800	14	
53	1.5	5200	5/3200	12	
54	1.5	5300	5/3600	10	

Relation of the apparatus capacity and quantity of filtered wine during filtration of red wine “Dzvelshavi” are shown in Fig. 4.

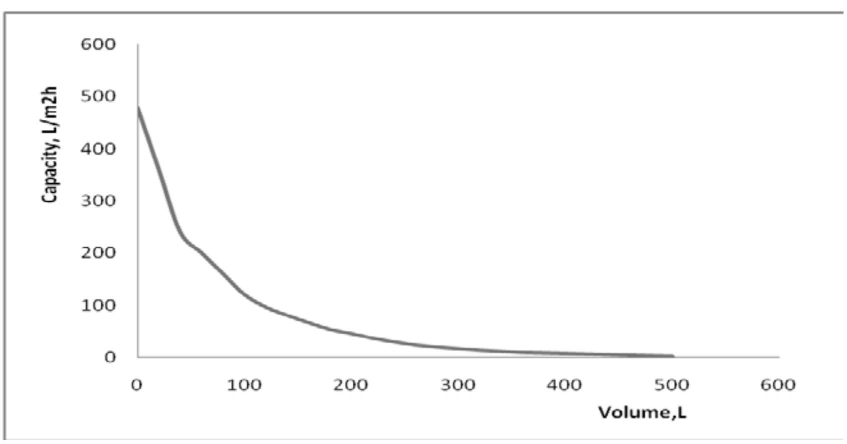


Figure 4. Relation of the apparatus capacity and quantity of filtered wine during filtration of red wine ”Dzvelshavi“

After restoration of apparatus capacity we performed filtration of 200 L of wine produced from “Tsolikauri” grapes from the family of Suliko Baramidze residing in Zendidi village, Keda region. Microfiltration apparatus purified the wine. As a result of were shown that the filtrate was clean from suspended substances, besides quantities of all the compounds dissolved in the wine were the same as in the initial wine (before filtration). The results of changes of apparatus capacity are shown in Table 8 here below.

Table 8. The results of filtration of red wine ”Tsolikauri“.

№	Working pressure, P atm.	Q-ty of filtered wine, l	Filtrate q-ty, l/sec.	Apparatus capacity, $l/m^2.h$	Remark
1	0.8	initial	1/15	480	wine
2	0.8	20	1/16	450	temperature
3	0.8	40	1/18	400	7^0C
4	0.8	60	1/20	360	
5	0.8	80	1/25	288	
6	0.8	100	1/30	240	
7	0.8	120	1/36	200	
8	0.9	140	1/45	160	
9	0.9	160	1/60	120	
10	0.9	180	1/75	96	
11	0.9	200	1/90	80	

The relation of the apparatus capacity and quantity of filtered wine in filtration of white wine produced from “Tsolikauri” is shown in graphical scheme (Fig. 5).

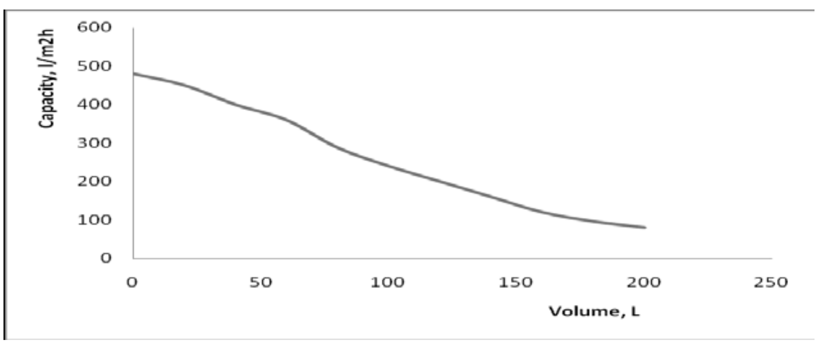
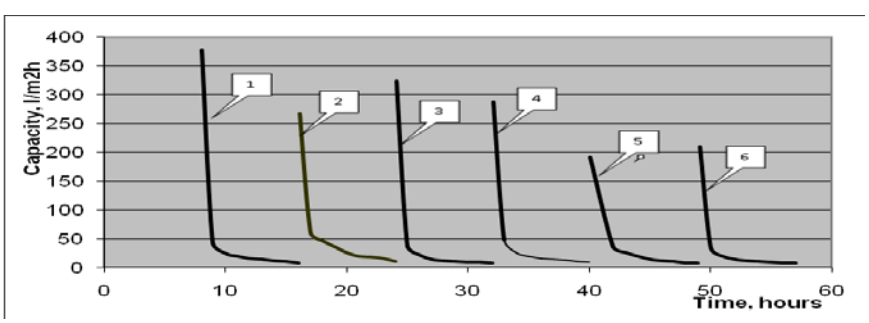


Figure 5. Relation of the apparatus capacity and quantity of filtered wine during filtration of white wine ”Tsolikauri“

Changes of apparatus capacity in filtration of various wines is shown in Figure 6.



Time, hours

Figure 6. Changes of apparatus capacity in filtration of various wines

Curve 1. Wine N 16 “Manavis Mtsvane”; initial capacity Q=378 l/m^2h
Curve 2. Wine N 42 “Rkatsiteli”; initial capacity Q=268l/m^2h
Curve 3. Wine N 33 “Rkatsiteli”; initial capacity Q=324 l/m^2h
Curve 4. Wine N 11 “Rkatsiteli”; initial capacity Q=288 l/m^2h
Curve 5. Wine N 23 “Tsolikauri”; initial capacity Q=192 l/m^2h
Curve 6. Wine N 55 “Rkatsiteli”; initial capacity Q=210 l/m^2h

We performed filtration of various liquid foods up to complete clogging of membranes in the experimental microfiltration apparatus, i.e. up to maximum reduction of capacity and researched the process of membrane regeneration.

Our microfiltration apparatus is fit for wine production, as it is designed for running regenerants by both direct and reverse flow. Besides, its design allows disassembling, washing of membranes and assembling. This process is so simple, that any winery employee can operate it. In spite of the mentioned above, our goal includes restoration of capacity of membranes in microfiltration apparatus without disassembling of apparatus and with use of such chemical agents that may be completely washed out of the apparatus for its further use.

In all cases we washed the clogged membranes with drinking water, then with sodium hydroxide NaOH; nitric acid - HNO_3; acetic acid - $C_2H_4O_2$; hydrochloric acid - HCI; hydrogen peroxide - H_2O_2; sodium carbonate - Na_2CO_3; sodium bicarbonate - $NaHCO_3$; solution of sodium hypochlorite – NaOCI. The results are shown in Table 9.

Table 9. Indexes of restoration of microfiltration membranes clogged in wine filtration

№	Reagents	Reagents concentration, %	Initial capacity of apparatus before clogging, $l/m^{2\cdot}h$	Capacity of apparatus after clogging, $l/m^{2\cdot}h$	Apparatus regeneration time, min.	Apparatus capacity after regeneration, $l/m^{2\cdot}h$
1	H_2O	-	480	10	30	50
2	NaOH	1 5 10	480	50 76 90	30 30 30	76 90 90
3	C_2H_5OH	1 5 10	480	90 90 90	30 30 30	90 90 90
4	HNO_3	1 5 10	480	90 90 90	30 30 30	90 90 90
5	$C_2H_4O_2$	1 5 10	480	90 90 90	30 30 30	90 90 90
6	HCI	1 5 10	480	90 120 130	30 30 30	120 130 130
7	H_2O_2	1 5 10	480	130 130 150	30 30 30	130 150 150
8	Na_2CO_3	1 5 10	480	150 150 150	30 30 30	150 150 160
9	Na_2CO_3	1 5 10	480	160 160 160	30 30 30	160 160 180
10	NaOCI	1 5 10	480	180 200 380	30 30 30	200 380 450

The results prove that the best extent of regeneration of membranes clogged with wine is reached in case of use of sodium hypochlorite solution as a regeneration reagent. Correspondingly, further we performed regeneration of membranes clogged in filtration of various wines with solution of sodium hypochlorite. In further regeneration we researched dependence of concentration of sodium hypochlorite solution on capacity growth indexes and we researched time of complete expulsion of reagent from the apparatus with drinking water after completion of regeneration.

Regeneration of membranes clogged in wine filtration was especially hard after filtration of red wine produced from "Dzvelshavi" grapes, as apart suspended substances the wine red pigment was found on membrane surface. The results of regeneration of membranes with the regenerant (NaOCl) are given in Table 10.

Table 10. Results of regeneration of membranes clogged in filtration of red wine"Dzvelshavi"

№	Reagents	Reagents concentration, %	Initial capacity of apparatus before clogging, l/m^{2}h	Capacity of apparatus after clogging, l/m^{2}h	Apparatus regeneration time, min.	Apparatus capacity after regeneration, l/m^{2}h
1	drinking water running	_	450	2	30	5
2	NaOCI	1	450	5	30	100
		5		100	30	220
		10		220	30	350
		20		350	30	430
3	drinking water running	_	450	430	30	430
4	H_2O_2	1	450	430	30	430
		5		430	30	440
		10		440	30	450
5	drinking water running	_	450	450	30	450

As is shown in Table 10, for economic use of regenerants, at the fist stage of regeneration of clogged membranes we have to remove suspended substances from membrane surface with drinking water flow (when we run regenerant through the clogged membrane, it got so polluted that we had to prepare the new portion of regenerant and run it again).

Although we increased concentration of sodium hypochlorite solution up to 20% but we did not achieve complete regeneration of membranes. For this purpose we used hydrogen peroxide additionally. As for regeneration of membranes clogged in filtration of white wines, for their complete regeneration 5-10% solution of sodium hypochlorite was enough, depending on extent of clogging of membranes surface and pores.

Based on the experimental results, we can conclude that in membrane filtration process of alcoholic drinks in wineries is required correct selection of the reagents for regeneration of clogged membranes providing their multiple use and for improvement of technological processes of regeneration. We selected reagents for regeneration of clogged membranes in wine filtration in wineries and two methods of regeneration.

Namely, at the first stage of restoration of microfiltration apparatus after filtration of white wine the surface of membranes were washed with waste waters, then with 5-10% solution of sodium hypochlorite (percentage of solution of sodium hypochlorite depends on extent of membrane clogging). Experimental data shows that for 0.5 m^2 working space of clogged membrane 10 L of 5-10% solution of sodium hypochlorite was needed. After the time required for complete washing of sodium hypochlorite out of microfiltration apparatus were fixed. It was established that for washing of sodium hypochlorite out of the membranes with waste waters 30 minutes is enough.

Technological process of regeneration of membranes clogged in filtration of red wine were added to the regeneration of membranes with hydrogen peroxide, i.e. after washing with waste waters (at the first stage) we carried out regeneration with 5-10% solution of sodium hypochlorite, with waste waters, with 5% solution of hydrogen peroxide and finally with waste waters during 30 minutes. Technological process of regeneration of the membranes clogged in filtration of wine is complicated and multistage. Namely, we rinse by water the apparatus without pressure during 30 minutes to purify the membrane surface. At the following stage we wash it with 5% solution of sodium hypochlorite in circular mode without pressure during 30 minutes and finally - with waste waters up to complete removal of sodium hypochlorite during 20 minutes. After that the apparatus is ready for filtration.

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