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Study of the Use and Reuse of Activated Charcoal in Rum Processing

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INTRODUCTION

The removal of specific amounts of rum components by activated charcoal, by reason of its adsorptive capacity, has been reported in the literature (*1,2*).[2] This paper presents the work done by the authors to evaluate the adsorptive capacity of six different brands of charcoal commercially available, and to determine the highest possible efficiency in their use for rum processing.

MATERIALS AND EQUIPMENT

Six brands of vegetable active commercial charcoal and a mixture of two of them were used. They were identified from CC-1 to CC-7. Charcoal identified as CC-4 is a mixture of CC-1 and CC-2 in a 2:1 ratio.

Also, a filtering system with a 10- to 25-gallon-per-hour capacity was used. The filtering membrane consisted of cotton filter tubes on polypropylene cores. A liter-capacity mixing apparatus was used to prepare the filtering medium.

The samples used in this work were experimental rums produced at the Rum Pilot Plant and aged for 4, 3, and 1½ years, respectively, in oak barrels. These samples were transferred to stainless steel barrels to prevent further change in their composition while this study was in progress.

EXPERIMENTAL PROCEDURE

Seven different experiments were performed in this study. The objectives of each were as follows:

[1] Technical Assistant in Chemistry, Technical Director and Chemist Collaborator, respectively. The authors wish to express their gratitude to Mrs. Milagros Montalvo and Jaime Rosario, Research Assistants, for their collaboration in the furfural and esters analysis, respectively; also to Mr. Cruz Aguilera, and A. Belardo Yadós, Associate Chemists, for their aid in the preparation of the manuscript. They were members of the scientific personnel of the Rum Pilot Plant of the Agricultural Experiment Station, Mayagüez Campus, University of Puerto Rico, Río Piedras, P. R., at the time this work was done.

[2] Italic numbers in parentheses refer to Literature Cited, p. 18.

EXPERIMENT 1.—To determine the capacity of various doses of different charcoals to remove congeners and color from rums.

EXPERIMENT 2.—To determine the minimum contact time between charcoal and sample required for maximum removal of specific components.

EXPERIMENT 3.—To compare the effectiveness of a single total dose addition *vs.* an incremental dose addition.

EXPERIMENT 4.—To study the effects of two consecutive charcoal treatments.

EXPERIMENT 5.—To determine the removal capacity of a given charcoal in rums aged for different periods of time.

EXPERIMENT 6.—To study reactivation procedures for used charcoals, and to determine the efficiency of the reactivated charcoals with respect to the removal of tannins and color.

EXPERIMENT 7.—To study the removal of copper and iron from rums by charcoal treatment.

The general procedure in treating a sample with charcoal was the following: One gram of paper pulp was mixed throughly for 1 minute in a mixing apparatus with 1 liter of rum. This suspension was filtered by means of the filter in order to provide a coating on the filter itself. Two liters of the rum to be treated were mixed with the charcoal being evaluated for the required length of time, and then filtered through the pulp-covered filter. The first 500 ml. of filtrate were discarded. The remainder was collected and analyzed. Total filtration time was approximately 5 minutes.

In experiment 1 the removal capacity of congeners and color from rum was studied for five different charcoals and a mixture of two of these charcoals. Four different doses of each charcoal were employed and the rum used in this experiment was analyzed after each treatment and compared with the analysis of the untreated rum. Doses of 1.5, 2.5, 3.5, and 4.5 g./liters of rum were used. The tabulated results are presented in table 1.

To determine the minimum contact time between charcoal and sample required for maximum removal of congeners and dolor charcoal CC-1 at a dose of 1.5 g./liter was employed. The general procedure was followed and periods of contact time of 1, 3, 5, 8, and 15 minutes were studied. The contact time referred to is the time during which the charcoal is mixed in the mixing apparatus with the rum being treated. It does not include the filtration time. Table 2 and figures 1 and 2 present the results of this experiment.

Charcoal CC-3 in a dose of 3.5 g./liter was used in experiment 3 to compare the effect of an incremental dose *vs.* that of a single total dose added in one step. The incremental treatment consisted of adding charcoal CC-3 to 2 liters of rum at a dose of 1 g./liter. After 1 minute of mixing, the rum was filtered and to the filtrate a second dose of 2.5 g./liter was added. In this manner the total dose of 3.5 g./liter was applied in two increments. The single-dose treatment of 3.5 g./liter was applied to a different 2-liter

TABLE 1.—*Removal of congeners and color in a heavy-type rum, using commercialy available activated charcoal (CC) nos. 2, 3, 4, 5, 6, and 7*

Congener	Original sample	Mg. of congener per 100 ml. of rum at 86° P.[1]																							
		Charcoal dose 1.5 g./liter—and percentage removal by—						Charcoal dose 2.5 g./liter—and percentage removal by—						Charcoal dose 3.5 g./liter—and percentage removal by—						Charcoal dose 4.5 g./liter—and percentage removal by—					
		cc 2	cc 3	cc 4	cc 5	cc 6	cc 7	cc 2	cc 3	cc 4	cc 5	cc 6	cc 7	cc 2	cc 3	cc 4	cc 5	cc 6	cc 7	cc 2	cc 3	cc 4	cc 5	cc 6	cc 7
Acidity	43	2	5	5	16	14	18	7	6	6	23	24	25	9	8	7	28	33	30	9	8	7	32	38	35
Aldehydes	14	1	1	2	0	0	0	1	1	1	0	0	0	1	2	1	0	0	0	1	2	3	0	0	0
Esters	36	2	2	0	8	8	0	2	2	0	11	8	0	2	2	0	14	8	0	2	2	0	14	5	0
Furfural	.36	19	19	14	22	28	19	28	25	22	36	39	25	42	31	28	44	47	28	47	44	33	53	58	39
Fusel oil	36	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Tannins	20	35	40	40	46	35	32	50	55	60	61	50	42	60	75	68	67	59	54	65	81	76	71	66	61
Color[2]	.35	54	50	53	58	37	30	68	65	76	78	57	43	80	85	92	84	69	57	86	92	91	87	77	68

[1] Degree proof wherever used.

[2] Optical density wherever used.

sample following the general procedure. The results of this experiment are presented in table 3.

The application of a double dose of charcoal in two consecutive treatments was studied in experiment 4, using charcoal CC-1. The rum sample was treated with a 4.5-g./liter dose and the filtrate obtained was again treated with a 4.5-g./liter dose of the same charcoal. Table 4 and figures 3 and 4 show the results obtained in this experiment.

In experiment 5 three types of rum (light, medium, and heavy) were treated with different doses of a mixture of charcoals CC-1 and CC-2 in a 2:1 ratio (identified as CC-4) to determine whether there was any difference in the removal capacity of these mixtures of charcoals with rums of different periods of aging. Charcoals CC-1 and CC-2 were used in this ratio

TABLE 2.—*Removal of congeners and color in a heavy-type rum at different intervals of time, using activated charcoal No. 1*

Congener	Mg. of congener removed per 100 ml. of rum at 86° P.[1]										
	Original sample	Concentration after 1 minute	Percentage removal	Concentration after 3 minutes	Percentage removal	Concentration after 5 minutes	Percentage removal	Concentration after 8 minutes	Percentage removal	Concentration after 15 minutes	Percentage removal
Acidity	68	65	4	65	4	65	4	65	4	65	4
Esters	100	94	6	93	7	93	7	93	7	92	8
Furfural	.37	.34	8	.34	8	.34	8	.34	8	.34	8
Tannins	26	12	53	12	53	12	53	12	53	12	53
Color[2]	.38	.18	52	.17	55	.17	55	.17	55	.16	57

[1] Degree proof wherever used.

[2] Optical density, wherever used.

since this mixture is currently used in the Rum Pilot Plant for rum processing. Table 5 and figures 5 and 6 present the data obtained in this experiment.

The reactivation of used charcoals was studied to determine the possibility of recovering charcoals after using them in the treatment of rums. Four different procedures for reactivation were studied in this experiment:

1. Washing the used charcoal with distilled water at room temperature and filtering.
2. Washing the used charcoal with distilled water followed by flushing the mixture with steam for 15 minutes and filtering while hot. Temperature was 98° C.
3. Direct heating in a muffle furnace at 100°, 200°, and 300° C.
4. Direct heating in a muffle furnace at 300° C. for different periods of time.

The charcoal used in this reactivation study was CC-4, the mixture of CC-1 and CC-2 in a 2:1 ratio. Enough used charcoal was obtained and sub-

divided in portions for use in the different reactivation procedures. Using the direct heating procedure at 300° C. for 30 minutes, a charcoal mixture was sucessively reactivated 4 times. After each reactivation period the

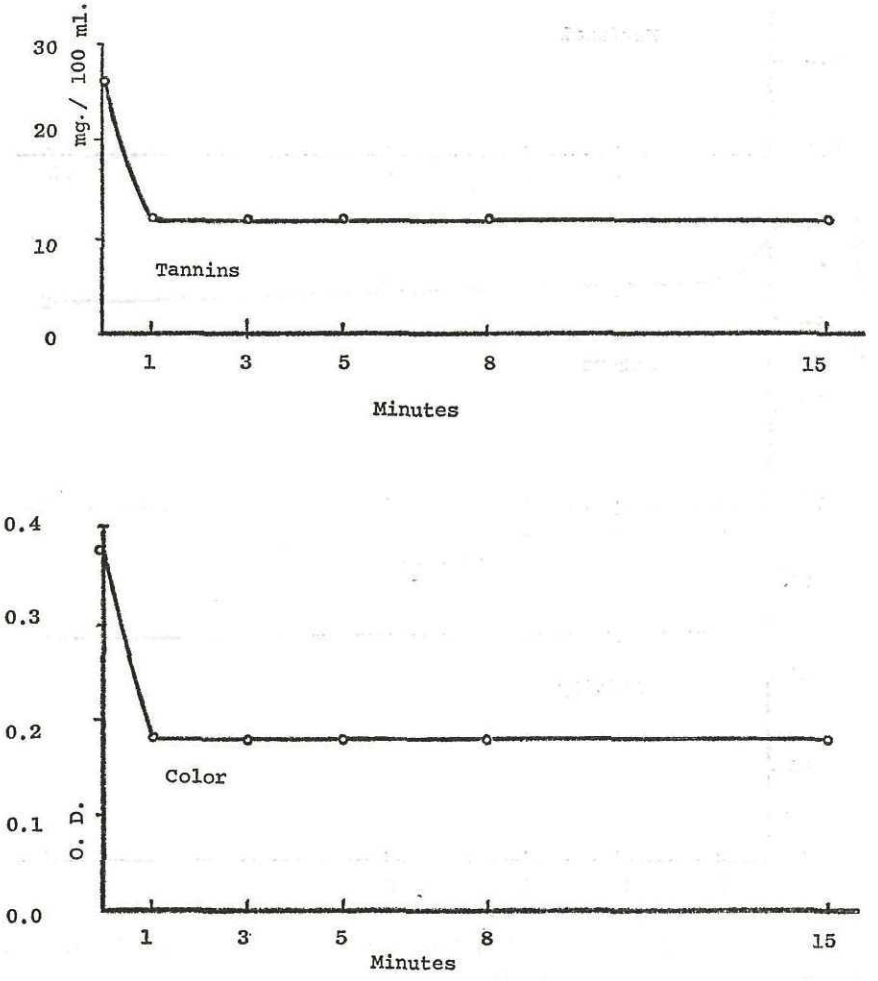

FIG. 1.—Removal of congeners in a heavy type of rum at different intervals of time using activated charcoal No. 1.

charcoal was used in the treatment of the rum sample in order to find out whether the efficiency was altered by the reactivation. Tables 6, 7, 8, and 9, and figures 7 and 8 show the results obtained in this experiment.

Experiment 7 was carried out to study the feasibility of removing copper and iron from rums. The rum sample used was a 1:1 mixture of two Puerto

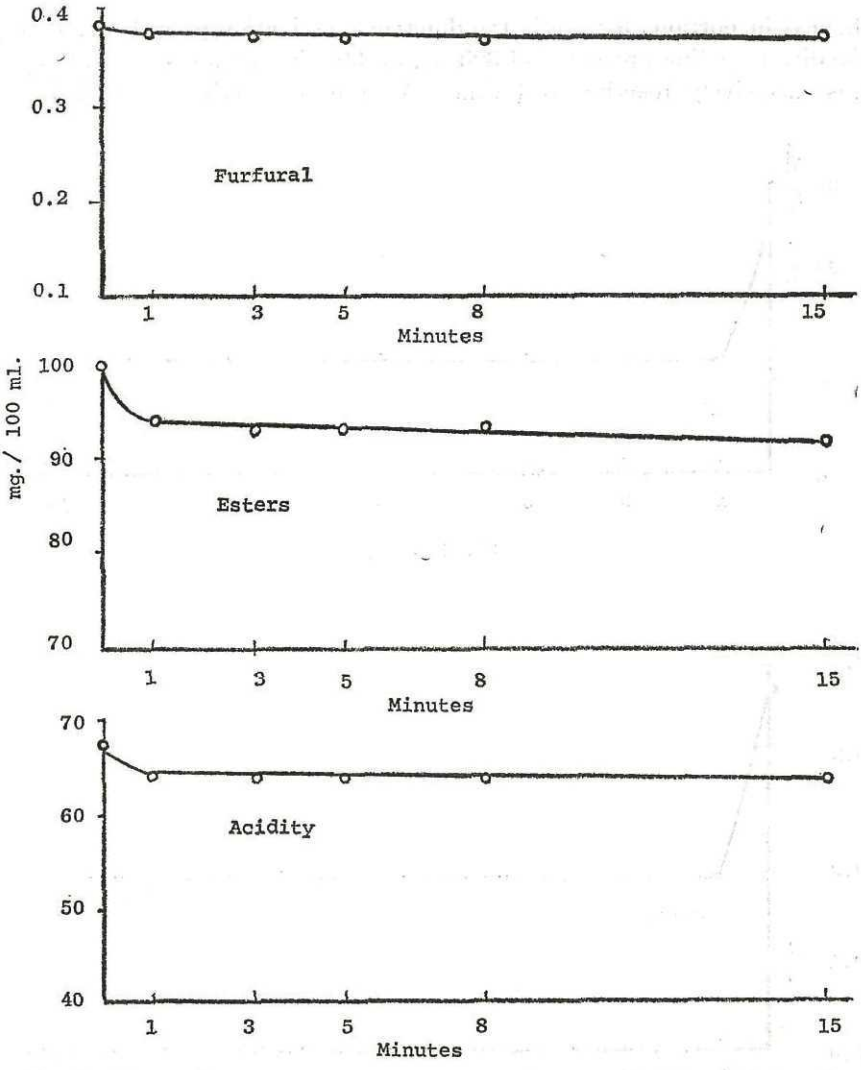

FIG. 2.—Removal of congeners in a heavy type of rum at different intervals of time using activated charcoal No. 1.

Rican rums of 1965 which had been found to contain the highest concentrations of copper and iron of all the Puerto Rican rums of that year. This mixture contained 0.5 p.p.m. of iron and 1.12 p.p.m. of copper. Charcoal CC-4 was found to remove 79 percent of the copper content and 76 percent of the iron content of the sample.

TABLE 3.—*Congener and color removal in a heavy-type rum*[1]

Congener	Mg. of congener per 100 ml. of rum at 86° P.[2]		
	Original sample	Incremental dose 1.0 + 2.5 g./liter	Single total dose 3.5 g./liter
		Removal	
Acidity	71	6	6
Aldehydes	19	5	5
Esters	99	7	5
Furfural	.43	37	35
Fusel oil	84	0	0
Tannins	27	75	69
Color[3]	.38	79	76

[1] *Incremental dose vs. single total dose of charcoal CC-3.*
[2] Degree proof wherever used.
[3] Optical density, wherever used.

TABLE 4.—*Congener and color removal in a rum sample by successive treatments with charcoal CC-1*

Congener	Mg. of congener per 100 ml. of rum at 86° P.[1]				
	Original sample	First treatment—dose 4.5 g./liter		Second treatment—dose 9.0 g./liter	
		Concentration after treatment	Percentage removal	Concentration after treatment	Percentage removal
Acidity	42	40	5	40	5
Aldehydes	14	14	0	14	0
Esters	38	36	5	34	10
Fusel oil	38	37	0	36	0
Furfural	.33	.21	36	.12	64
Tannins	21	3.5	83	1.1	95
Color[2]	.34	.02	94	.004	99

[1] Degree proof wherever used.
[2] Optical density wherever used.

The rum samples were analyzed before and after charcoal treatment by the official analytical methods of the Rum Pilot Plant (*3*).

RESULTS

Table 1 presents a comparison of the removal capacities of the five activated charcoals studied. Removal capacity is highest for tannins, furfuraldehyde, and color. Aldehydes and esters are only slightly removed.

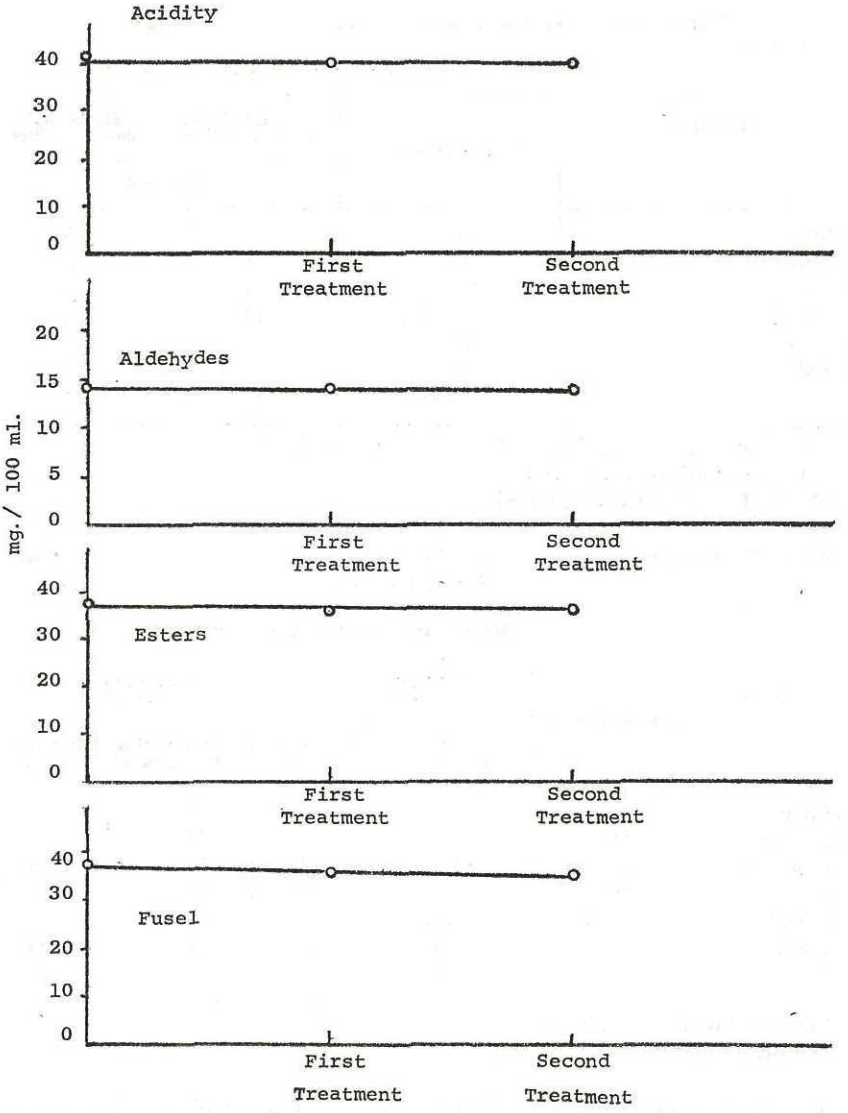

FIG. 3.—Congener and color removal from a rum sample by successive treatments with charcoal cc-1.

Fusel oil is not removed by the charcoals studied. A larger charcoal dose removes a larger amount of some components, such as tannins and furfuraldehyde, other congeners like aldehydes and fusel oil remain unaltered. The removal of color is also increased by a larger dose. In general, char-

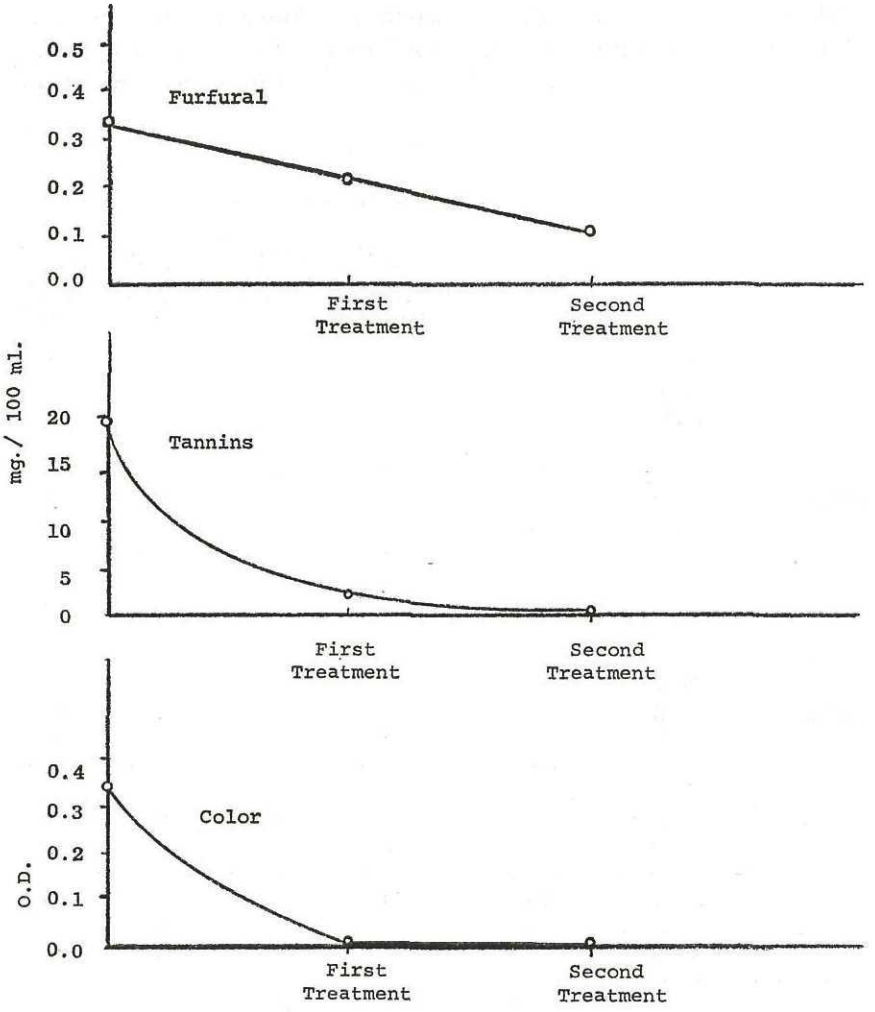

FIG. 4.—Congener and color removal from a rum sample by successive treatments with charcoal CC-1.

coals CC-3 and CC-5 behave similarly to charcoal CC-4 used at present at the Rum Pilot Plant.

Table 2 and figures 1 and 2 present the results obtained in the determination of the minimum time of contact between the rum and the charcoal necessary to get the maximum color and congener removal. It was found that a 1-minute contact period is adequate. Charcoal CC-1 was used in this study.

The treatment of a rum with an incremental dose of charcoal instead of with a dose added all at once does not increase the removal efficiency of the charcoal (see table 3). The treatment of a rum sample with equal

TABLE 5.—*Removal of congeners and color in 3 types of rums using varying doses of charcoal CC-4*[1]

Congener	Mg. of congener per 100 ml of rum at 86° P.[2]					
	Type of rum	Original sample	Charcoal dose 1.5 g./liter—and percentage removal	Charcoal dose 2.5 g./liter—and percentage removal	Charcoal dose 3.5 g./liter—and percentage removal	Charcoal dose 4.5 g./liter—and percentage removal
Acidity	Heavy	43	5	6	7	7
	Medium	21	8	9	9	9
	Light	10	0	0	0	0
Aldehydes	Heavy	14	2	1	1	1
	Medium	4	0	0	0	0
	Light	4	0	0	0	0
Esters	Heavy	36	0	0	0	0
	Medium	13	0	0	0	0
	Light	4	0	0	0	0
Furfural	Heavy	.36	14	22	28	33
	Medium	.14	14	21	29	36
	Light	.07	28	43	57	57
Fusel oil	Heavy	36	0	0	0	0
	Medium	10	0	0	0	0
	Light	5	0	0	0	0
Tannins	Heavy	20	40	60	68	76
	Medium	15	57	74	81	88
	Light	6	83	86	92	95
Color[3]	Heavy	.35	53	76	92	91
	Medium	.25	74	91	93	95
	Light	.08	88	88	95	95

[1] A mixture of charcoals CC-1 and CC-2 in a 2:1 ratio.
[2] Degree proof throughout.
[3] Optical density wherever used.

consecutive doses of a charcoal does not alter significantly the amounts of congeners and the color removed from that rum, as indicated by the data in table 4 and in figures 3 and 4.

Table 5 and figures 5 and 6 present the removal capacity of different doses of a mixture of charcoals CC-1 and CC-2 for light, medium, and heavy

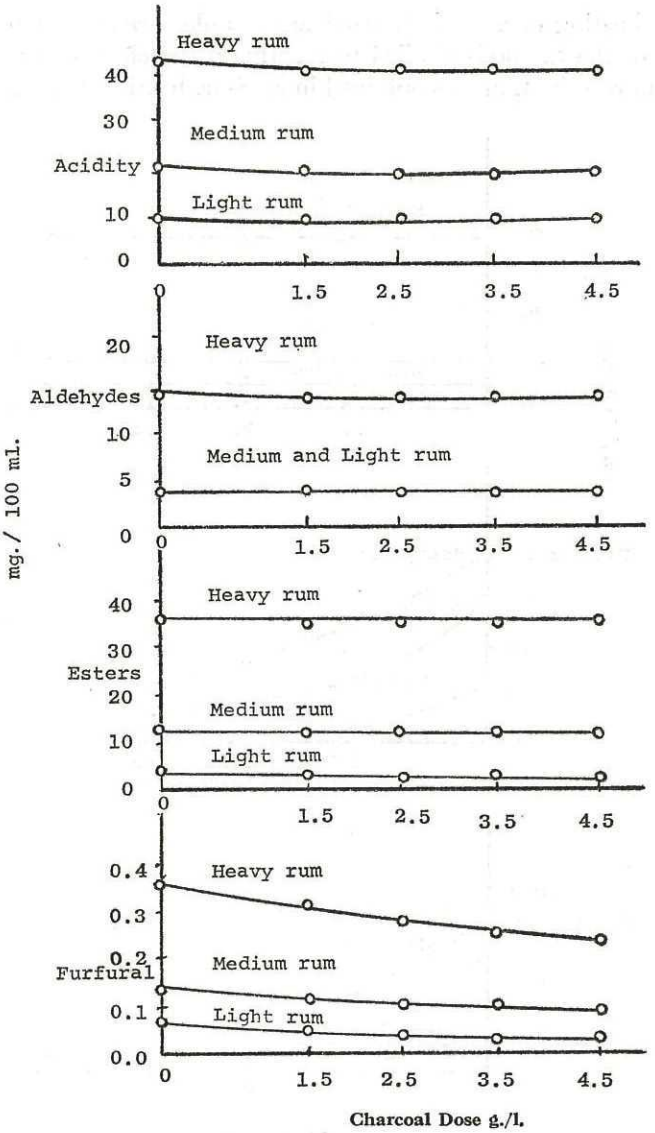

FIG. 5.—Removal of congeners in 3 types of rums using varying doses of charcoal CC-4.

rums. These results confirm the usefulness of increasing the charcoal dose, especially in the case of heavy rums. There is no significant increase in percentage of congener removal for doses greater than 2.5 g./liter in the light and medium rums.

Direct heating of a used charcoal in a muffle furnace was found to be the best of the methods studied to reactivate the charcoal (see table 6). Maximum reactivation was obtained in an 8-hr. heating period at 300° C.

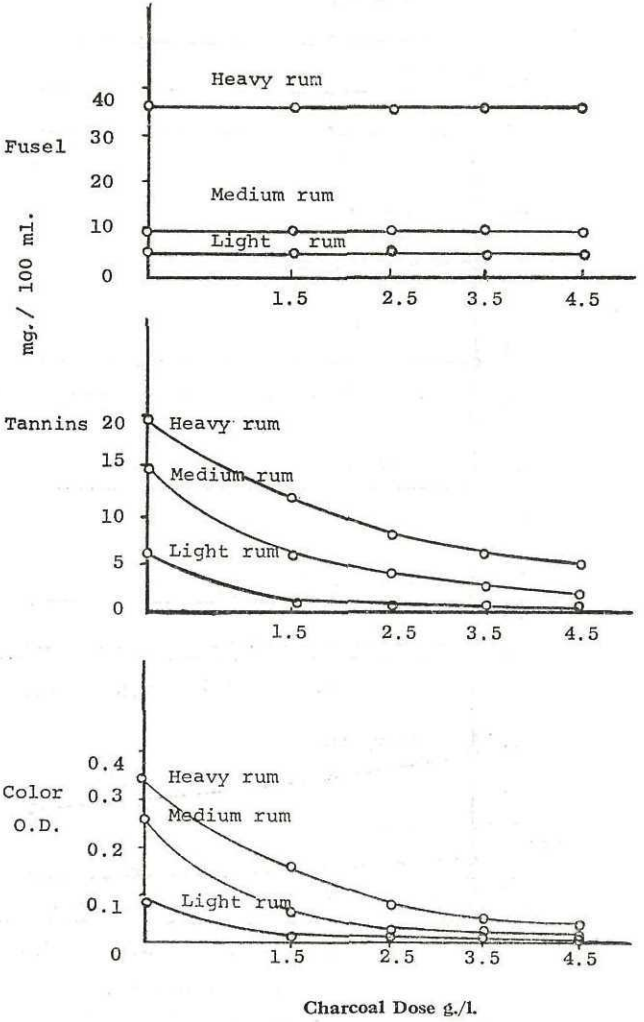

FIG. 6.—Removal of congeners in 3 types of rums using varying doses of charcoal CC-4.

However, a ½-hr. heating period at 300° C. is adequate. A charcoal so treated acquires 79 percent of its original color-removal capacity, and 78 percent of its original tannin-removal capacity. The results are presented in tables 7 and 8 and in figure 7. A sample of charcoal CC-4 was used and

TABLE 6.—*Reactivation of a used charcoal (CC-4) as determined by the removal of color and tannins*

Charcoal sample used in the rum treatment	Color			Tannins		
	Optical density	Percentage removal	Percentage reactivation	Mg./100 ml.	Percentage removal	Percentage reactivation
No charcoal used (rum without treatment)	0.45			25		
Original charcoal	.11	76		12	52	
Used charcoal without reactivation	.34	24	32	20	20	38
Used charcoal reactivated by heating at 100° C.	.30	33	44	19	24	46
Used charcoal reactivated with water	.37	18	24	19	24	46
Used charcoal reactivated with water followed by by heating at 100° C.	.39	13	17	19	24	46

TABLE 7.—*Reactivation of a used charcoal (CC-4) as a function of temperature, as determined by the removal of color and tannins from a rum*

Charcoal samples used in the rum treatment	Temperature of reactivation	Color of the rum			Tannins in the rum		
		Optical density	Percentage removal	Percentage reactivation	Mg./100 ml.	Percentage removal	Percentage reactivation
No charcoal used (rum without treatment)	—	.36			24		
Original charcoal	—	.12	67		11	54	
Used charcoal without reactivation	—	.32	11	16	19	21	39
Used charcoal reactivated by heating at	100° C.	.26	28	42	18	25	46
Used charcoal reactivated by heating at	200° C.	.20	44	66	16	33	61
Used charcoal reactivated by heating at	300° C.	.16	56	84	13	46	85

reactivated repeatedly by this method without a significant decrease of the removal capacity. After the fourth heating the color-removal capacity was 88 percent of the original and the tannin-removal capacity was 74 percent. (See table 9 and figure 8).

CONCLUSIONS

The results obtained in this study indicate that, in general, the charcoals used have very similar capacities for the removal of congeners and

TABLE 8.—*Reactivation of a used charcoal as a function of time, as determined by the removal of color and tannins from a rum using a mixture of CC-1 and CC-2*

Charcoal sample used in the rum treatment	Time of reactivation (hours)	Color of the rum			Tannins in the rum		
		Optical density	Percentage removal	Percentage reactivation	Mg./100 ml.	Percentage removal	Percentage reactivation
No charcoal used (rum without treatment)	—	.36			24		
Original charcoal	—	.12	67		11	54	
Used charcoal without reactivation	—	.32	11	16	19	21	39
Used charcoal reactivated by heating at 300° C.	¼	.20	44	66	18	33	61
Do.	½	.17	53	79	14	42	78
Do.	1	.17	53	79	13	46	85
Do.	2	.16	56	84	13	46	85
Do.	8	.15	58	87	12	50	93

TABLE 9.—*Percentage removal of tannins and color, and percentage of reactivation with respect to tannins and color of a 2:1 mixture of charcoals CC-1 and CC-2 (charcoal CC-4)*[1]

Mg. of congener per 100 ml. of rum at 86° P.

Procedure	Color			Tannins		
	Optical density	Percentage removal	Percentage reactivation	Mg./100 ml.	Percentage removal	Percentage reactivation
Original sample, not treated	0.26			18		
Sample treated with fresh charcoal mixture	.10	62		9	50	
Sample treated with used charcoal mixture, not reactivated	.25	8	13	16	11	22
Sample treated with charcoal mixture, after 1st. reactivation	.13	50	81	11	39	78
Sample treated with charcoal mixture once used after 1st. reactivation	.24	12	19	15	17	34
Sample treated with charcoal mixture after 2nd. reactivation	.12	54	87	10	44	88
Sample treated with charcoal mixture once used after 2nd. reactivation	.22	15	24	14	22	44
Sample treated with charcoal mixture after 3d. reactivation	.14	50	81	10	44	88
Sample treated with charcoal mixture once used after 3d. reactivation	.23	12	19	14	22	44
Sample treated with charcoal mixture after 4th. reactivation	.15	46	74	10	44	88

[1] Reactivation procedure: Heating at 300° C. for half-hour; dose-1.5 q./liter; rum used, RPP Experimental, heavy type.

color in rums. Charcoals CC-2, CC-3, CC-4, and CC-5 have some advantage over charcoals CC-6 and CC-7 in the removal of color. Charcoals show maximum output when reactivated by direct heating in a muffle furnace. By this process a charcoal sample was reactivated and reused consecutively

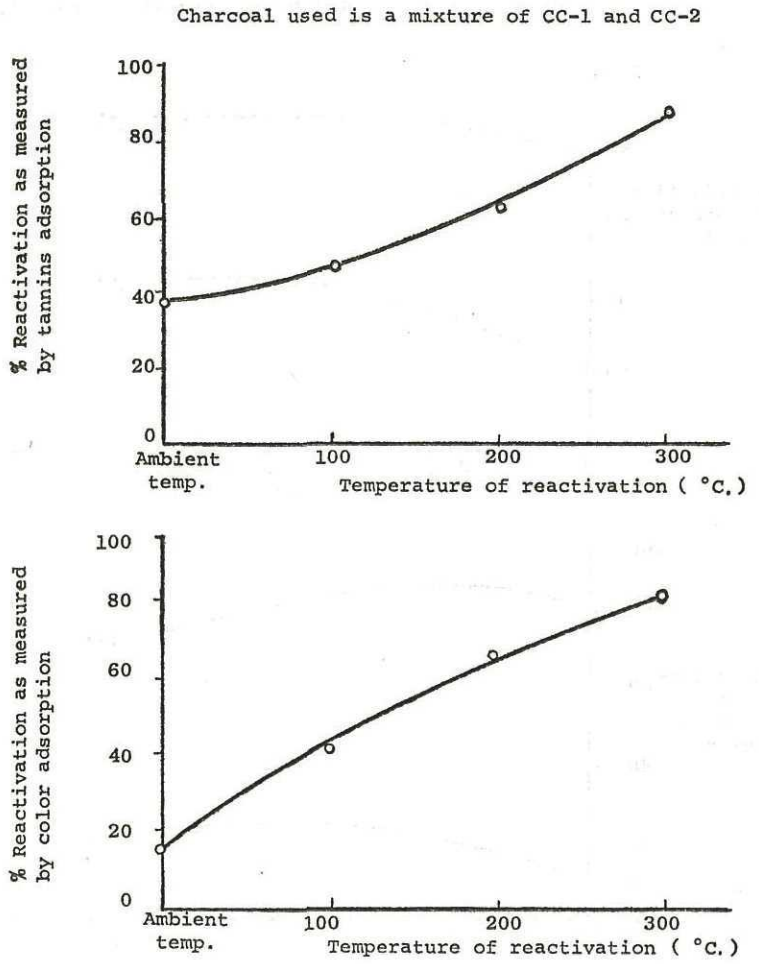

FIG. 7.—Reactivation of a commercial charcoal as a function of temperature at time of reactivation: 30 minutes.

four times without a significant loss in its capacity for removing color and tannins.

SUMMARY

Activated charcoals are used in rum-processing for the specific removal of detrimental components such as tannins and furfuraldehyde, and for

the elimination of excessive color. Other rum components are also removed in this process in varying degrees.

In this study five commercial brands of activated charcoals were evalu-

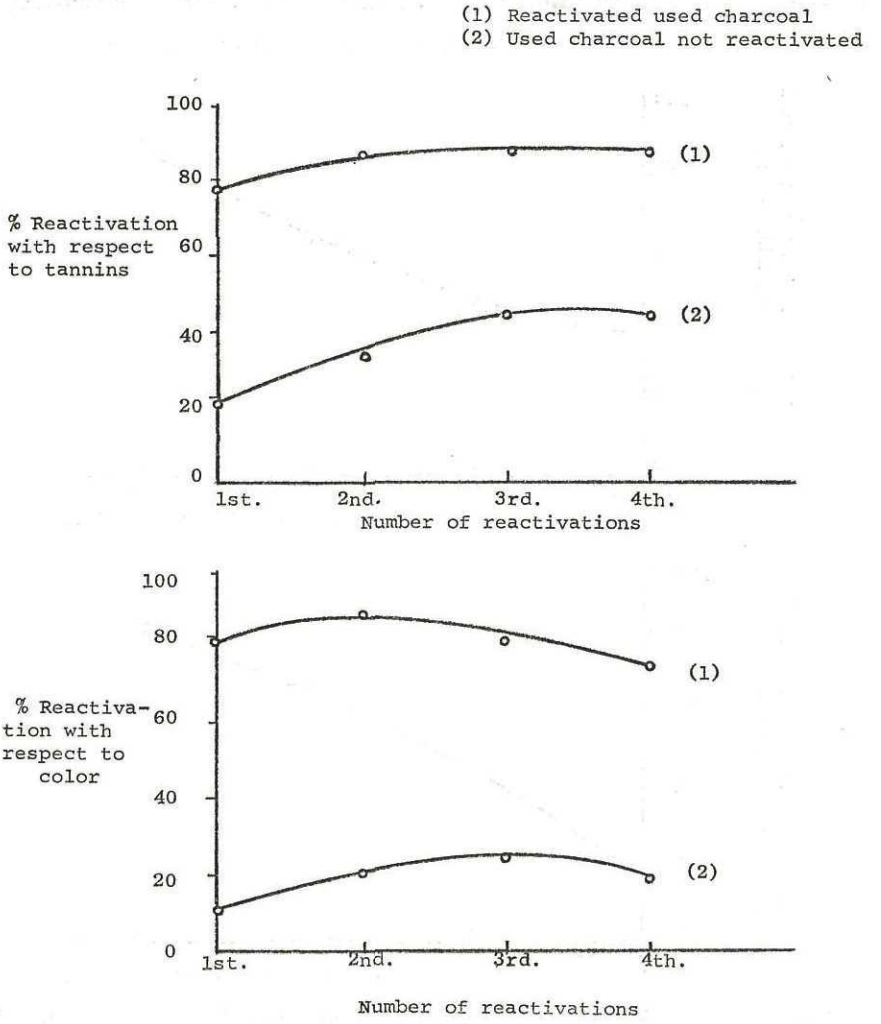

FIG. 8.—Percentage of reactivation of a used charcoal with respect to tannins and color *vs.* number of successive reactivations.

ated to determine their removal capacity for different rum components. Efforts were made also to determine the best way to use and reactivate charcoals to attain their maximum efficiency in rum-processing.

In the study of the best ways of utilizing the charcoal the following aspects were investigated: The capacity of various doses of different char-

coals in removing congeners and color from rums; the effect of two consecutive charcoal treatments; the effectiveness of adding a single total dose *vs.* an incremental dose; the minimum contact time between charcoal and sample; the removal capacity of a given charcoal in rum aged for different periods of time.

In general the charcoals used have very similar capacities for the removal of congeners and color in rums. Charcoals CC-2, CC-3, CC-4, and CC-5 have some advantages over charcoals CC-6 and CC-7 in the removal of color.

The following methods for reactivating the used charcoal were studied: washing the used charcoal with water, washing the used charcoal with water followed by flushing the mixture with steam; and direct heating at different temperatures. The reactivation by direct heating at 300° C. for 30 minutes was found to be the best. By this process a charcoal sample was reactivated and reused consecutively four times without a significant loss in its capacity for removing color and tannins.

The data are presented by means of tables and graphs. A discussion of the results is included.

RESUMEN

El carbón activado se usa en la elaboración de los rones para eliminarles los componentes perjudiciales, como son los taninos y el furfural, y para quitarles el color excesivo. Durante este proceso, también se extraen en mayor o menor grado otros componentes.

En este estudio se evaluaron cinco carbones activados comerciales para determinar su capacidad de remover los diferentes componentes del ron. Se trató, además, de determinar el mejor modo de emplear y reactivar el carbón para obtener la máxima eficiencia en la elaboración de los rones. Los resultados se presentan en una serie de tablas y gráficas incluyéndose, además, una discusión sobre los mismos.

En el estudio de la mejor utilización del carbón los aspectos siguientes fueron estudiados: El uso de varias dosis de distintos carbones para remover congenéricos y color; el uso de dos dosis consecutivas; el efecto de usar una dosis total o parcial; el tiempo mínimo de contacto entre el carbón y la muestra de ron; la capacidad de remover congenéricos y color dependiendo de la edad del ron.

En general los carbones estudiados tuvieron capacidades similares para remover congenéricos y color en el ron. Los carbones CC-2, CC-3, CC-4 y CC-5 presentan ventajas sobre los carbones CC-6 y CC-7 para remover color.

Para reactivar el carbón usado se estudiaron métodos de lavado con agua, lavado seguido de vapor directo y calentamiento a distintas temperaturas. La reactivación calentando en una mufla a 300 °C. por 30 minutos resultó la más ventajosa, pudiéndose por este proceso reactivar 4 veces

consecutivas un mismo carbón sin una pérdida significativa de la capacidad de remover color y taninos.

Los resultados se presentan en una serie de tablas y gráficas y se incluye una discusión de los mismos.

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