Forest Systems
29 (1), eSC02, 7 pages (2020)
eISSN: 2171-9845
https://doi.org/10.5424/fs/2020291-15829
Instituto Nacional de Investigación y Tecnología Agraria y Alimentaria (INIA)

SHORT COMMUNICATION

OPEN ACCESS

Optimum cork stopper diameter for a proper wine sealing performance when modifying bottleneck diameter: a first approach

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Abstract

Aim of the study: This study present a theoretical model that allow establishing the proper relationship between forces and diameters that take part in sealing for ensuring an adequate closure during storage time, and obtained the optimum stopper diameter for a proper sealing performance when modifying bottleneck diameter.

Area of study: The proposed model is of interested to the whole cork value chain from forest owners to natural cork stoppers manufacturers.

Materials and methods: The optimum cork stopper diameter depends mainly on stopper quality and the compression rate applied in the bottling operation. In this study, we establish the stopper diameter when reducing bottleneck diameter, applying a compression rate of 33% when corking, and for natural cork stoppers which quality allows to recover its initial diameter to 96% after 24 h since compression.

Main results: For a bottleneck diameter of 18 mm, the value of the stopper diameter should be at least of 22.3 mm, and for a bottleneck diameter of 17 mm, the value of the stopper diameter should be at least of 20.3 mm.

Research highlights: These results try to solve one of the main worries of natural cork stopper manufacturers, which is the scarcity of raw cork suitable for manufacturing them. However this study is also of interested to forest owners because the increment of cork suitable for natural cork stoppers manufacturing means an increment in cork value.

Key words: bottling; corking; compression force; compression rate; diameter recovery; relaxation force; relaxation ratio.

Abbreviations used: *Ds* (Cork Stopper Diameter); *Dg* (Caliper Diameter the Corking Machine); *Db* (Bottleneck Diameter); *Dr* (Recovered Diameter); *Fc* (Compression Force); *Fr* (Relaxation Force); *CR* (Compression Rate); *RR* (Relaxation Ratio); *RD* (Diameter Recovery).

Authors' contributions: Conception and design of experiment: MSG, FGH, CP. Acquisition of data: MSG, FGH. Analysis and interpretation of data: MSG, FGH, CP. Supervision and coordination of the research: MSG, CP. Revision and drafting of the manuscript; MSG, CP

Citation: Sánchez-González, M., González-Hernández, F.; Prades, C. (2020). Optimum cork stopper diameter for a proper wine sealing performance when modifying bottleneck diameter: a first approach. Forest Systems, Volume 29, Issue 1, eSC02. https://doi.org/10.5424/fs/2020291-15829.

Received: 03 Oct 2019. **Accepted:** 05 Mar 2020.

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Funding Agencies/Institutions
The authors received no specific funding for this work, made under the collaboration agreement CC13-045 between INIA-CIFOR and the University of Córdoba.

Competing interests: The authors have declared that no competing interests exist.

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Introduction

Wine sealing is an important step of the wine making process. Its main priority is maintain wine quality during bottling, storage and shipping. Premium quality wines have traditionally been packed mainly in glass bottles sealed with natural cork stoppers. Glass, as an inert material, assures maximum impermeability towards oxygen, while cork stoppers foster a good evolution of wines by allowing slow migration of oxygen (Caillé *et al.*, 2018). Natural cork stopper is the most valuable product that can be manufactured with high quality cork.

The main physical-mechanical variables dealing with a good sealing performance are: stopper density, the compression force required to compress stopper diameter to caliper closure diameter, the relaxation force exerted by the stopper against the glass surface after inserted into the bottle, the diameter recovery evolution after compression and the extraction force the final consumer must applied to extract the stopper (González-Hernández *et al.,* 2014; Sánchez-González & Pérez-Terrazas, 2018). During the bottling operation, the stopper is inserted into the bottleneck by compressing it with a caliper. Therefore, there is a close relationship among stopper diameter, bottleneck diameter and caliper closure diameter. The stopper dimensions were settled dependent on the standardized inner dimensions of the bottleneck, in 24 x 44 mm as the optimal stopper dimensions for sealing most of still wines. Regarding caliper closure diameter, this is related with stopper diameter through the compression rate that is the ratio between the uncompressed stopper diameter and compressed diameter. A good practice when bottling consists in not applying compression rates above 33 % (Sánchez- González & Perez-Terrazas, 2018), because of the negative effect it has on the elasticity, the diametric recovery and the relaxation force exerted by the fitted stopper in the bottleneck.

In order to produce natural cork stoppers of 24 mm, it is needed that harvested cork planks have a thickness of more than 27 mm. The reason is that the stopper axis is parallel to the cork oak stem axis. In a cork harvest, the percentage of cork suitable for natural cork stoppers manufacturing is very variable ranging from 35 % to 60 % (Rives *et al*., 2012; Demertzi *et al.,* 2016). In addition, cork production is decreasing due to lack of regeneration and ageing of cork oak stands (Pasalodos-Tato *et al*., 2018). However, the demand for natural cork stoppers has increased which is reflected in the evolution of natural cork stoppers exports in world leader countries in in the cork sector, Portugal and Spain (APCOR, 2018).

Given this situation, it is worthy to consider the possibility of reduce stopper diameter in order to increase the quantity of cork suitable for natural cork stopper manufacturing, but maintaining or improving the requirements for a good sealing performance. For this purpose is needed to modify stopper diameter and bottleneck diameter. The main aim of this study is to establish the proper relationship between forces and diameters that take part in sealing for ensuring an adequate closure during storage time. We assumed that a proper closure is achieved when the relaxation ratio (González-Hernández *et al.,* 2014), remains constant irrespective of the values of the aforementioned diameters. Based on that assumption, we intend to obtain the optimum stopper diameter for a proper sealing when reducing bottleneck diameter.

Material and methods

A sample of natural cork stoppers were used to determine the optimum stopper diameter for a proper sealing when reducing bottleneck diameter. To do so, in a first step it was obtained which values should have the coefficient *k* for a proper sealing when using cork stoppers of high quality using bottlenecks of different diameters, by the execution of two mechanical tests under standard bottling conditions, a compression test and a relaxation test. In a second step, and assuming as the optimal sealing conditions the standard ones, i.e. cork stoppers of 24 mm of diameter fitted in bottlenecks of 19 mm of dimeter, the stopper diameter was calculated.

Sampling

A randomly selected batch of 500 one-piece natural cork stoppers of the first commercial quality and nominal dimensions of 24x44 mm were sent to the INIA-CIFOR cork laboratory from a Spanish cork stopper manufacturer. A sample of 35 cork stoppers selected with the criteria of having similar values of density were used. This criteria was applied to try to play down the influence of stopper density in its mechanical properties (Anjos *et al*., 2008; Anjos *et al*., 2014; Rosa & Fortes, 1988). The sample of 35 natural cork stoppers were randomly subsampled in 7 groups with 5 stoppers per group, corking each group using different diameters of the tube that simulate bottleneck. Those diameters were 17, 18, 18.4, 19, 20, 21 and 22 mm.

Cork stopper were acclimatized at 20 °C and 65 % of relative humidity. Stabilization was considered to have been achieved when the weight variation in two consecutive weighings was less than 0.024 g (which is equivalent to a humidity difference of 0.1 %). Once acclimatized, stoppers were weighed and measured using Mitutoyo ID-F150 digital vernier calipers. Room conditions during the measures were an ambient temperature of 20 ºC ± 4 ºC and relative humidity of 50% ± 10%. Stoppers density was calculated as already reported in González-Hernández *et al*. (2014).

Mechanical tests

The biaxial compression force (Fc) in the modelling sample set was measured using a semiautomatic corking machine equipped with a load cell (UTILCELL, Mdo: 650 SNo 460775(02) Emax:

2Tn), as already reported in González-Hernández *et al*. (2014). Cork stoppers were inserted in each bottleneck tube by applying the same compression rate of 33 %.

The diameter recovery evolution of each cork stopper was assessed by measuring the stopper diameter 24 hours after the test with Mitutoyo ID-F150 digital vernier callipers.

The relaxation force (*Fr*) (González-Hernández *et al.,* 2014) in the both sample set was measured by inserting each bottleneck tube with the stopper inserted within it into the device developed in the INIA-CIFOR Cork Laboratory (González-Hernández *et al.,* 2012) as already reported in González-Hernández *et al*. (2014).

Statistical analysis

For each measured variable, the mean, standard deviations, minimum and maximum were calculated. In a preliminary analysis, the assumptions of normality, independence and homogeneity of the variance were verified. The hypothesis of no differences among stopper included in each subsampled group was tested using ANOVA. The differences were examined using pairwise comparisons according to the Tuckey test. All tests were conducted at the α=0.05 level and all analyses were carried out using the SAS software version 9.4 (SAS Institute Inc., 2016).

Results and discussion

Theoretical model

The strain-stress curves described for cork by Fortes *et al.* (2004) and Mano (2002) are very similar to those that take place during corking. In the formers, the compression load is done in a unique axial direction, while during corking the cork stopper is subjected to a concentric compression. Fig. 1 shows a schematic depiction of the evolution of the stress-strain hysteresis loop during corking, sealing period and uncorking.

During corking, the cork stopper diameter is compressed from its initial value (*Ds*) to the caliper diameter of the corking machine (*Dg*). The stress applied increases to a maximum value of compression (*Fc,* point C in Fig. 1) describing the loading curve 0C. At point C, when the cork stopper is fitted in the bottleneck of diameter (*Db*), the stress is reduce from the compression force (*Fc,* point C in Fig. 1) to the relaxation force (*Fr,* point H in Fig. 1) and simultaneously the diameter of the cork stopper is partly recovered from *Dg* to *Db*, describing the unloading curve CH. When the cork stopper is fitted in the bottleneck the diametric recovery goes on describing the whole unloading curve until reaching the maximum value of the recovered diameter (*Dr*) that the stopper will reach as long as the wine is bottled.

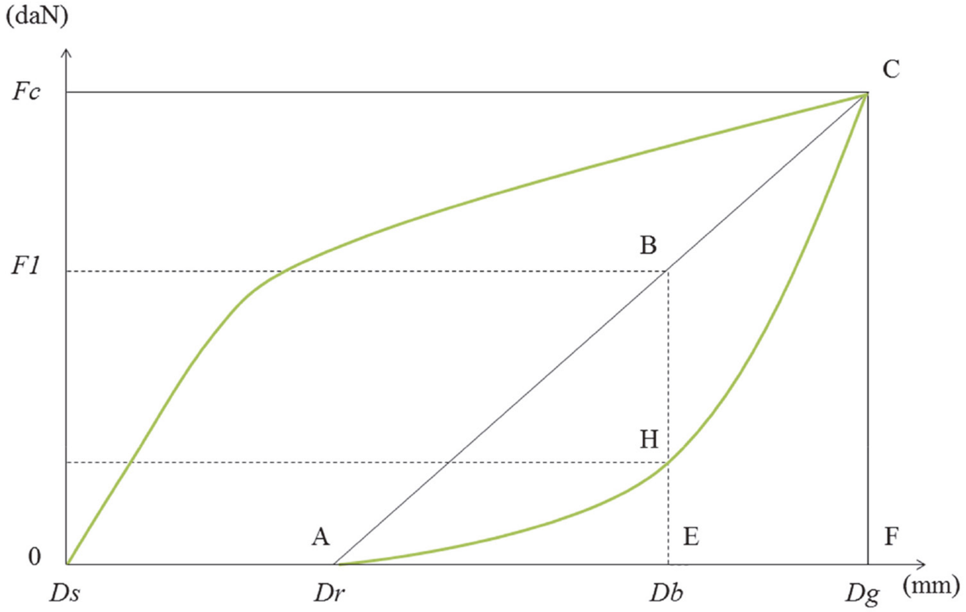

Figure 1. Schematic depiction of the evolution of the stress–strain hysteresis loop during corking, sealing period and uncorking. *Ds*: Stopper diameter. *Dr*: Recovered diameter. *Db*: Bottleneck diameter. *Dg*: Caliper diameter of the corking machine. *Fc*: Maximum compression force. *F1*: Compression force when the cork stopper is fitted in the bottleneck.

The similarity of triangles *ACF* and *ABE* allows getting to:

$$\frac{Fc}{\left(Dr-Dg\right)}=\frac{F1}{\left(Dr-Db\right)} \tag{1}$$

Therefore, $F1=\frac{\left(Dr-Db\right)}{\left(Dr-Dg\right)}Fc$ (2)

For a given value of bottleneck diameter (*Db*), the values of *Fc* and *F1* can be related through a coefficient $k=\frac{Fr}{F1}$ (3)

Replacing *k* in (2) we get to: $Fr=k\frac{\left(Dr-Db\right)}{\left(Dr-Dg\right)}Fc$ (4)

If we take into account that the recovered diameter (*Dr*) can also be expressed as a function of the diameter recovery (*RD*) and the caliper closure diameter can also be expressed as a function of the compression rate (*CR*). Then equation (4) can be expressed as a function of those variables:

$$Fr=k\frac{\left(RD\cdot Ds-Db\right)}{\left(RD\cdot Ds-\left(\left(1-CR\right)\cdot Ds\right)\right)}Fc \tag{5}$$

Where *Fr* is the relaxation force; *Fc* is the compression force; *k* is the relaxation coefficient, *Ds* is the stopper diameter, *Db* is the bottleneck diameter, *Dr* is the recovered diameter, *Dg* is the caliper closure diameter, *RD* is the diameter recovery and *CR* is the compression rate. The relationship between Fr and Fc can be expressed by means of the relaxation ratio (*RR*) (González-Hernández *et al.* 2014):

$$RR=\frac{Fr}{Fc} \tag{6}$$

Then, $k=\frac{RR}{\frac{\left(RD\cdot Ds-Db\right)}{\left(RD\cdot Ds-\left(\left(1-CR\right)\cdot Ds\right)\right)}}$ (7)

Where *k* values depends on the quality of cork stoppers, the compression rate applied when corking, the bottleneck diameter and the stopper diameter. If we were able to determine which values should have the coefficient *k* for a proper sealing when using cork stoppers of a given quality, applying the same compression rate and using different bottlenecks diameter, we could establish the proper stopper diameter for each bottleneck diameter by applying the following equation obtained from equations (5) and (6):

$$Ds=\frac{k\cdot Db}{RR-RR\cdot RD-RR\cdot CR+k\cdot RD} \tag{8}$$

The stopper diameter depends mainly on the bottleneck diameter (*Db*), the compression rate (*CR*) and the stopper quality, because the relaxation ratio (*RR*) and the diametric recovery (*RD*) depends on stopper quality and the compression rate applied in the bottling operation (Anjos *et al.*, 2008; Fortes *et al.*, 2004; González-Hernández *et al.*, 2014).

Optimum stopper diameter when reducing bottleneck diameter

The statistics values for each measured variable are shown in Table 1. ANOVA indicated no significant differences in biaxial compression force (*Fc*) values among subsample groups (F value = 1.92 p value = 0.1772). This result was expected because the same compression rate was used. The compression force takes mean values of 228.91 daN, between 193.80 and 261.00 daN (Table 1). These values are very similar to those obtained by previous studies (Beorlegui, 2014; González-Hernández *et al.,* 2014; Prades *et al*., 2014) for natural cork stopper of the same quality which is expected since measurement was taken in the same way using a clamp or caliper which means that are values of biaxial compression.

The maximum relaxation force (*Fr*) applied by each stopper against the bottleneck tube, and measured using the device developed in the INIA-CIFOR Cork Laboratory (González-Hernández *et al.,* 2012), shows significant differences among subsampled groups of stoppers (Table 1 and Fig. 2) except for 18.4 mm which mean values is not significantly different from the man value for 18 mm and 19 mm. As expected, the relaxation force decrease as the bottleneck diameter increase when using natural cork stoppers of 22 mm of diameter.

The diametric recovery (*RD*) after 24h from compression do not show significant differences among subsampled groups of stoppers. Since the same compression rate was applied in all of them, this result was also expected. The mean value for the whole modelling sampled set was 95.929 % ranging from 94.539 % to 97.433 %. Then, it can be considered that after 24h after compression to 33 % the tested natural cork stoppers recovered 96 % of its initial diameter. In Fig. 1, *Dr* refers to the value of the recovered diameter that cork stopper would reach 24 h after being fitted in the bottleneck, we are going to consider that this diameter is the 96 % of the stopper diameter. Following this assumption, we have calculated *k* values for the modelling sampled set (Table 1).

The relationship between bottleneck diameter and *k* follows a parabolic pattern (Fig. 3). The minimum point

Table 1. Characterization of tested variables in the modelling sample set (*Db*: bottleneck diameter; *min*: minimum; *max*: maximum; *std*: standard deviation).

	Compression force (daN)				**Relaxation force (daN)**			
Db	**mean**	**max**	**min**	**std**	**mean**	**max**	**min**	**std**
17	212.440	237.600	193.800	20.916	45.609	52.185	39.151	5.064
18	222.160	239.400	200.000	16.422	34.721	40.180	28.273	4.337
18.4	230.920	239.000	220.400	7.618	29.380	32.879	27.587	2.048
19	234.480	244.200	211.800	13.283	25.490	29.400	20.433	4.309
20	227.280	246.200	204.000	16.402	19.149	20.727	17.640	1.425
21	243.280	261.000	233.000	11.598	15.425	17.738	13.671	1.537
	Relaxation ratio				***Stopper density*** ($k \cdot m^{-3}$)			
Db	**mean**	**max**	**min**	**std**	**mean**	**max**	**min**	**std**
17	0.215	0.223	0.201	0.009	154.885	156.759	152.891	1.426
18	0.156	0.174	0.134	0.017	157.399	159.708	155.197	1.816
18.4	0.127	0.142	0.120	0.009	157.365	159.018	155.334	1.609
19	0.108	0.121	0.091	0.014	161.553	164.533	158.796	2.319
20	0.085	0.094	0.076	0.008	159.850	162.207	157.385	2.065
21	0.063	0.068	0.055	0.005	163.478	166.388	160.173	2.595
	Diametric recovery after 24h (%)				***k***			
Db	**mean**	**max**	**min**	**std**	**mean**	**max**	**min**	**std**
17	95.358	96.572	94.539	0.007	0.246	0.255	0.229	0.011
18	95.874	96.883	95.135	0.007	0.216	0.235	0.188	0.021
18.4	95.906	97.433	95.183	0.009	0.188	0.203	0.176	0.010
19	96.086	96.697	95.144	0.006	0.189	0.215	0.152	0.026
20	96.053	96.569	95.466	0.004	0.187	0.206	0.162	0.017
21	96.221	97.120	95.333	0.007	0.211	0.221	0.199	0.010
22	96.004	96.776	95.072	0.007	0.311	0.342	0.275	0.029

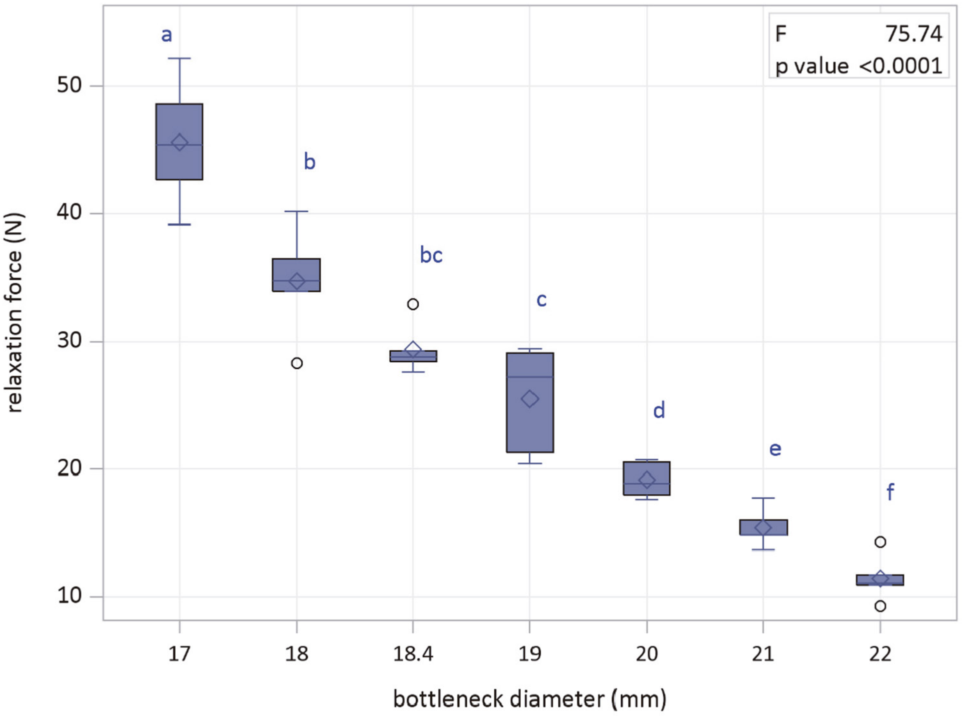

Figure 2. Boxplots of relaxation force distributions per subsampled group by bottleneck diameter. Boxplot notches indicate a 95 % confidence interval on the median. Different letters show significant differences ($p<0.05$).

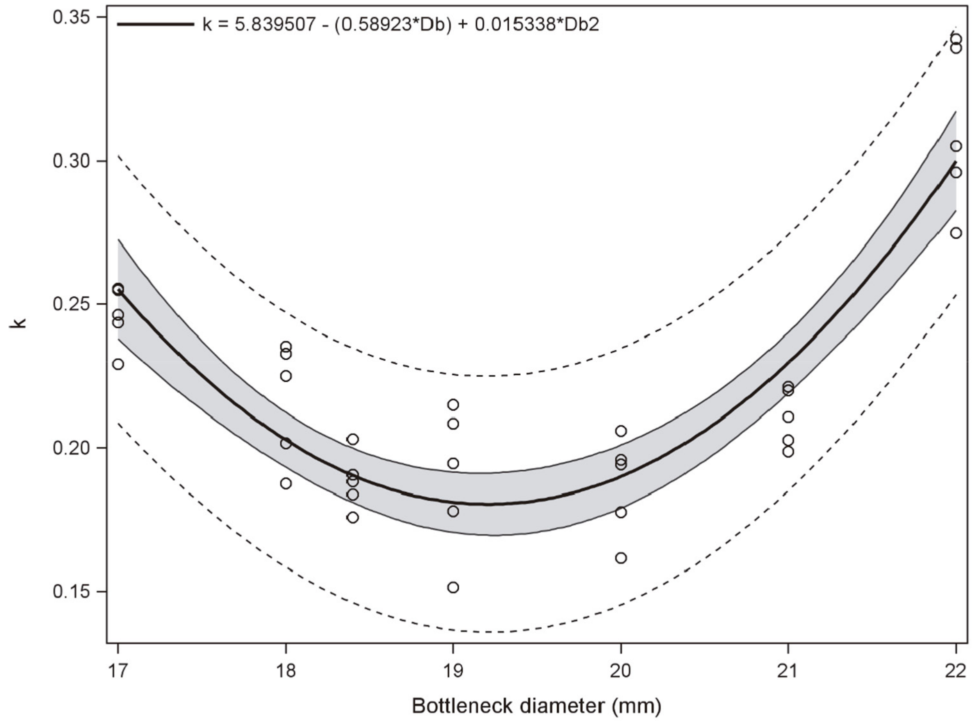

Figure 3. Variation of the k values calculated in the modelling sampled set with bottleneck diameter. Solid line represent the regression line. Dark-shaded region shows the 95 % confidence interval and dashed lines are upper and lower 95 % prediction intervals of the regression.

of the second degree parabola is reached at a bottleneck value of 19.2 mm, all the values of *k* above the minimum value corresponding to bottleneck dimeters smaller than 19.2 mm, can be considered as safe from a sealing performance perspective when the compression rate applied is 33 %. The equation shows in Fig. 3 jointly to equation (8) can be applied to estimate stopper diameter for a given bottleneck diameter smaller than 19 mm considering a compression rate of 33 % and, as relaxation ratio value, the mean value obtained for the sampled group of cork stoppers fitted in bottleneck tubes of 19 mm. Therefore, for a bottleneck diameter of 18 mm the value of the stopper diameter should be at least of 22.3 mm, and for a bottleneck diameter of 17 mm the value of the stopper diameter should be at least of 20.3 mm.

Conclusions

The theoretical model presented in this study allow to establish the proper relationship among stopper diameter, bottleneck diameter and compression rate for ensuring an adequate closure during storage time, and obtained the optimum stopper diameter for a proper sealing performance when modifying bottleneck diameter. In this study, we establish the stopper diameter when using bottleneck diameter of 17 and 18 mm, applying a compression rate of 33% when corking, and for natural cork stoppers which quality allow recovering a 96 % of its initial diameter 24 h after compression. These results are a first approach to one of the main worries of natural cork stopper manufacturers, which is the scarcity of raw cork suitable for manufacturing them. The proposed model must be validated in oncoming studies, applying this reduction, and considering both, cork stoppers quality and wine sealing performance. However these results are also of interested to forest owners because the increment of cork suitable for natural cork stoppers manufacturing means an increment in cork value.

Acknowledgements

The authors would like to thank the laboratory assistants Maria Luisa Cáceres Esteban and Lorenzo Ortiz Buiza for all their work in the laboratory, and to Adam Collins for his linguistic assistance.

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