

Proceedings in Engineering Mechanics
Research, Technology and Education

Lucas F. M. da Silva
Robert D. Adams *Editors*

6th International Conference on Adhesive Bonding 2021

Selected Contributions of AB 2021

Proceedings in Engineering Mechanics

Research, Technology and Education

Series Editors

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Lucas F. M. da Silva · Robert D. Adams
Editors

6th International Conference on Adhesive Bonding 2021

Selected Contributions of AB 2021

 Springer

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Preface

This volume is the first volume of the new book series *Proceedings in Engineering Mechanics—Research, Technology and Education*. This new series intends to publish the latest developments concerning fundamental research and applied technology of engineering mechanics. Education studies on science and technology are also the focus of this new series. The themes covered are very broad and have in common engineering mechanics. We want to go from fundamentals to applications and how these can be transferred to students.

This first volume of *Proceedings in Engineering Mechanics—Research, Technology and Education* contains selected papers presented at the 6th International Conference on Structural Adhesive Bonding (AB 2021), held in Porto (Portugal) during 8–9 July 2021 (www.fe.up.pt/ab2021). The goal of the conference was to provide a unique opportunity to exchange information, present the latest results as well as to discuss issues relevant to structural adhesive bonding research today. Approximately, 165 papers were presented by researchers from more than 27 countries.

In order to disseminate the work presented in AB 2021, selected papers were prepared which resulted in the present volume dedicated to adhesive bonding. A wide range of topics are covered resulting in 11 chapters dealing with the most recent research topics concerning adhesive bonding: adhesive formulation and properties (first four chapters), adhesion and surface treatments (three chapters), joint design (two chapters) and durability of structural adhesive joints (last two chapters). The book represents the latest trends in adhesive joining and serves as a reference volume for researchers and graduate students working with adhesive bonding.

The organizer and editor wish to thank all the authors for their participation and cooperation, which made this volume possible. Finally, I would like to thank the team of Springer-Verlag, especially Dr. Christoph Baumann, for the excellent cooperation during the preparation of this volume.

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Open Assembly Time of Water-Borne Polyvinyl Acetate (PVAc) Wood Adhesives Affected by Various Factors



Peter Bliem, Hendrikus W. G. van Herwijnen, Robert Meissl, Walter Stadlbauer, and Johannes Konnerth

Abstract Open assembly time is one of the most important processing parameters of water-borne polyvinyl acetate (PVAc) dispersion adhesives. Exceeding the open assembly time may cause a loss of bonding properties. It is essential to have knowledge about possible factors influencing the open time. The present work evaluates different influencing factors on open assembly time of PVAc adhesives. At first, a simple and highly reproducible method was developed to determine the open time. Subsequently, by using this method, open times at varying relative humidities, room temperatures and application rates of PVAc adhesives were investigated on absorbent substrate. As a general result, this method is suitable as an alternative for determining the open assembly time. Furthermore, clear effects of some parameters investigated could be observed. While an increase in relative humidity and adhesive application rate led to a prolonged open assembly time, an increase in room temperature showed only little negative effect. However, if wood substrate and adhesive temperatures are simultaneously increased with the room temperature, a severe shortening of the open time could be observed.

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Keywords Polyvinyl acetate (PVAc) · Open assembly time · Adhesives for wood

1 Introduction

A wide variety of adhesive systems are available for bonding wood. The selection of the appropriate adhesive is typically based on the bonding requirements and/or the desired properties of the bonded joint or on process-related characteristics. A general distinction can be made between thermosetting and thermoplastic adhesive polymers. Polyvinyl acetate (PVAc) is an important representative of thermoplastic adhesives and, along with thermosetting urea formaldehyde (UF) adhesives, a commonly used adhesive system in the furniture industry (Dunky 2003).

The mainly physically setting of the water-borne dispersion adhesives are easy in handling, physiologically harmless due to the absence of formaldehyde emission or other reactants, and capable of forming an adhesive film at room temperature. The synthesis of PVAc is a chain growth reaction of vinyl acetate monomers dispersed in water in the presence of an emulsifier (usually polyvinyl alcohol) by free radical initiators (Frihart 2013; Jaffe et al. 1990; Pizzi 1989). The mean length of the resulting polymer chains is up to 3 μm and it forms many hydrogen bonds with the wood substrate due to the high content of acetate groups and the flexible characteristic of the polymer backbone (Frihart 2013; Zeppenfeld and Grunwald 2005). PVAc sets by water penetration into the wood substrate and/or by evaporation of the water into the ambient air. In the process of setting, the polymer particles gradually come closer to each other until they form a homogeneous adhesive layer (i.e. film formation) (Dunky 2003; Frihart 2013). The so-called setting speed can be influenced by two main groups according to Theiling (1972) as well as Rabiej and Brown (1985). The first group represents the properties of the adhesive itself, depending on the formulation chosen by the adhesive manufacturer. Important factors are the type of polymer system, protective colloid system, solid content, particle size of the dispersion, viscosity of the adhesive, type and amount of modifiers. On the other hand, the adhesive user as the second crucial group, has an influence on the setting speed of the PVAc adhesives. Several important parameters in this regard are the absorptivity of the substrate, the material moisture content and ambient humidity, adhesive application quantity, open assembly time, wood density, temperature (adhesive, wood substrate, ambient air), annual ring position, proportion of early and late wood and many more. Some of these factors can be considered and controlled when processing PVAc adhesives. Due to production processes, the open assembly time is often a critical factor in the processing of these adhesives, as a certain amount of time is required from applying the adhesive until joining the adherends (Sedliacik et al. 2014). The open assembly time is defined as the time starting from applying the adhesive to the adherend(s) until the assembly of the joint without loss of bonding strength (EN 923 2016). In other words, it is the time in which the applied adhesive contains enough water to sufficiently wet the opposite substrate material. Exceeding the open time will result in a dried adhesive layer and thus to poor bondings (Frihart 2013).

How quickly the water from the PVAc adhesive penetrates into the substrate or evaporates essentially depends on the adhesive, application, ambient and material conditions. In literature, some factors influencing the open time are mentioned, but there is little information available on how strong the influence actually is. While an increase in substrate and ambient temperature as well as the porosity of the substrate is related to a reduction in open time, a decrease in adhesive application quantity, substrate moisture content as well as relative humidity is attributed to a decrease in open time (Habenicht 2009).

Thickeners such as hydroxyethyl cellulose, methylcellulose, polyvinyl alcohol or starches will increase viscosity when added to an PVAc adhesive and release water slowly (Jaffe et al. 1990). As a result, they can prolong open time. Humectants as hygroscopic substances (i.e. glycerine, various glycols, sorbitol, sucrose, urea) in PVAc adhesives keep the surface wet and extend the open time, but because they hold and reabsorb water to a certain extent, they will soften the material after drying (Jaffe et al. 1990).

The maximum open times specified in the technical data sheets of the adhesive manufacturers are measured under defined laboratory conditions. These standardized conditions cannot be reached consistently in the processing companies, leading to undesirable shorter open times and thus to inadequately bonded joints.

A standardized method for measuring open time is defined in the standard EN 16556, which involves gluing beech lamellas together and measuring the tensile shear strength from the resulting lap joint samples (EN 16556 2014). Furthermore, it is also mentioned that the determination of the open time can be done by single lap joint samples according to EN 205 Annex A. Tankut et al. (2016), for instance, measured the effect of different nanoparticles and open times on bonding strength by means of lap joint samples. Another method, which is particularly used in practice, is to check the surface tackiness of the applied adhesive by means of a simple finger test. Since these methods are time and resource intensive or very user-dependent and therefore not really reproducible, an alternative method for determining open time of water-borne PVAc adhesives has been developed within the present study. With this method, conclusions can be drawn more quickly about possible factors influencing the open time. In addition, high reproducibility can be achieved with this method, since the solid wood with the typical high natural variability is replaced by more homogeneous materials. In this work, it is hypothesized that relative humidity, ambient air, material and adhesive temperature as well as adhesive application quantity are critical factors on the duration of open time. Therefore, these factors were systematically varied and open times were measured using the newly optimized method. The aim was to gain a better understanding of the change in open time by varying the various parameters and how strongly they affect the open time.

Table 1 Overview of the PVAc glue characteristics according to the technical and safety data sheets

Parameter	Standard D3 PVAc	Standard D4 PVAc
Solid content (%)	50	50
pH	3.0	3.5
Density at 20 °C (g/cm ³)	1.08	1.09
Viscosity (mPa * s)	13000 Brookfield HB, spindle 3, 20 rpm, + 20 °C	5500 Brookfield HB, spindle 2, 20 rpm, + 20 °C
Organic solvent content (%)	2.2	1.8

2 Materials and Methods

In the first step, an alternative method for determining the open time of PVAc adhesives was developed. This method should be reproducible, easy in handling, with a maximum accuracy and provide prompt results. Subsequently, the open times at varying relative humidities, room temperatures and application rates of PVAc adhesives were investigated and analysed.

2.1 Adhesive System Characteristics

Two different industrial polyvinyl acetate glues were used in this study (H.B. Fuller Austria GesmbH, Wels, Austria). The main characteristics for both glues are listed in Table 1. Standard D3 PVAc fulfils the durability class D3 according to EN 204 (EN 204 2016) and is commonly used for veneering and surface/assembly gluing. The standard D4 PVAc is a one component PVAc glue offering improved water and heat resistance used for the production of window frames and doors (durability class D4).

2.2 Experimental Designs

In the first part of the present study, the influences of different environmental conditions, adhesive application quantities, and PVAc glue types on open time were investigated and Table 2 shows the experimental designs. The relative humidity was systematically changed, starting from 35 to 50% up to 65%. The ambient temperature of the testing room had two levels, namely 23 or 33 °C. The MDF (medium density fibre) board and adhesive temperature were always set to 23 °C. Therefore, MDF boards were first stored in standard climate and then tempered to the desired temperature in

Table 2 Experimental design with various possible influencing parameters for investigating open time

parameter			
relative humidity (%)	35 – 50 – 65		
ambient temperature (°C)	23 – 33		
MDF board and adhesive temperature (°C)	23	23 or 33 (corresponding to ambient temperature)	
glue application quantity (g/m ²)	105 – 135		
PVAc glue	standard D3 PVAc	standard D4 PVAc	standard D3 PVAc

a climate chamber shortly before use. PVAc glues, in contrast, were stored airtight inside the climate chamber at 23 °C.

In the second part of the work, the MDF board and D3 PVAc glue temperatures were also increased to 33 °C in case of an increased testing room temperature. The procedure for storing the boards and the adhesive was identical as described above, but now at 33 °C. Subsequently, the boards and the adhesive temperature corresponded to the ambient temperature of the testing room. This experiment was not conducted with the D4 PVAc in order to prevent premature reaction of the adhesive at correspondingly higher temperature.

A manual film applicator with gap clearances of 150 and 200 µm and a width of 60 mm was used for adhesive application (mtv messtechnik oHG, Erftstadt, Germany). Accordingly, these gaps resulted in adhesive quantities of 105 g/m² and 135 g/m², respectively.

2.3 Influence of Temperature on Viscosity

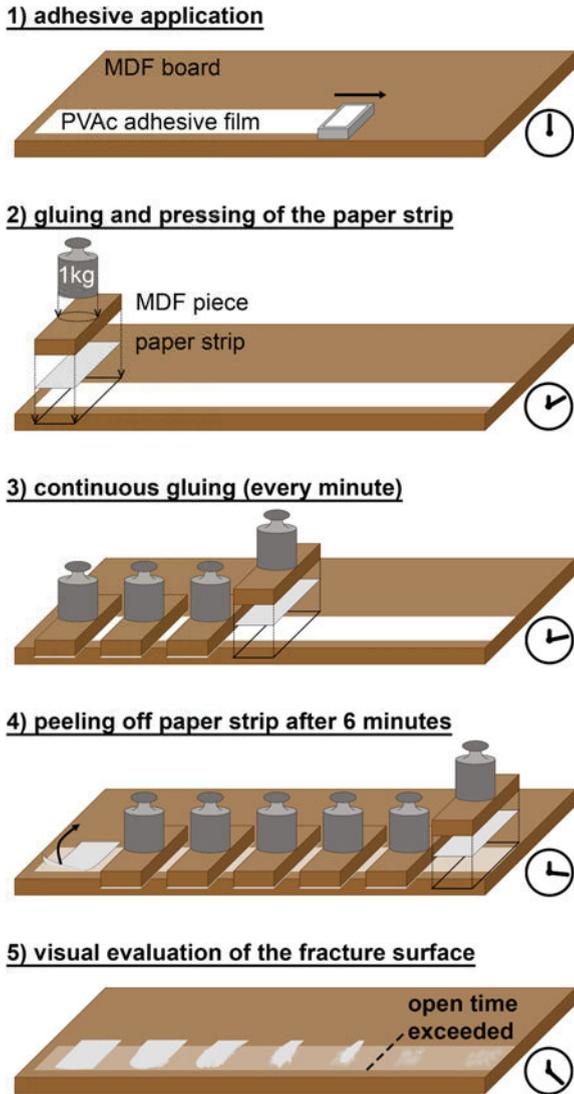
Since part of the study involved tempering the D3 PVAc glue to 33 °C, the viscosity of this glue was measured depending on the temperature. D4 PVAc glue was not tempered to 33 °C in the experiments, but a temperature ramp was also measured as additional information. Both temperature ramps were performed at testing temperatures between 22 and 34 °C at a constant shear rate of 10 1/s using a rheometer (MCR 302, Anton Paar GmbH, Austria) equipped with a cone-plate measuring system (CP 50–1, Anton Paar GmbH, Austria). Gap size was set to 0.101 mm resulting in a test volume of 0.57 ml.

2.4 Procedure for Determining the Open Time

The entire experiment was performed in a climate-controlled room exhibit a volume of 30 m³ at the technical centre of Wood K plus, Area Wood Materials Technologies (Tulln/Donau, Austria). Target air humidity and room temperature were set according to the experimental designs and monitored twice using a Sensirion digital sensor (SHTW2, Staefa, Switzerland) and a data logger (Lascar EL-USB-2+, Wiltshire, United Kingdom). The accepted fluctuation ranges of the measured climate conditions were $\pm 4\%$ for relative humidity and ± 2 °C for room temperature. During the individual measurements, the actual values were measured and used for the data evaluation.

Figure 1 shows the schematic procedure for measuring open time and is a development of an unpublished laboratory method used in the adhesive producing industry. In step 1, the tempered PVAc glue was applied on a likewise tempered MDF board (Kaindl MDF Standard E1E05, M.Kaindl OG, Wals, Austria) using a manual film applicator. Dimensions of the MDF board were thereby 600 × 200 × 19 mm (length × width × thickness) with an average board density of 700 kg/m³. All MDF boards for the individual experiments performed were previously stored at standard climate conditions (20 °C and 65% relative humidity) until constant mass was reached, resulting in an average moisture content of 8.6%. MDF board was tempered to the desired temperature (i.e. 23 or 33 °C) inside a separate climate chamber prior to adhesive application. The relative humidity inside the climate chamber has been slightly increased to keep the moisture content of the MDF board constant and prevent undesired drying or influence of variable substrate moisture content. Therefore, the relative humidities were set to 66% (at 23 °C) and 68% (at 33 °C) respectively. In addition, the weight of the MDF boards were measured before and after storage to determine any possible changes in the moisture content. The use of MDF instead of another wood-based material or solid wood seemed to be the best solution to avoid undesired influences of less homogeneous materials (i.e. density differences, fibre deviations, different permeability of the surface, etc.). The actual amount of glue applied was determined gravimetrically by means of a scale with an accuracy of 0.01 g. A few minutes were then waited so that the previously applied glue could dry off a little. Then a simple strip of copy paper, as described below, was placed on the glue film and weighted down with a piece of MDF (out of the same material as the MDF board) and a weight of 1 kg (step 2). The dimensions of the paper strip and the MDF piece were 120 × 45 mm (length × width), with an overlapping area of these on the MDF board of 90 × 45 mm. This resulted in a specific pressure of 2421 Pa. Copy paper with a grammage of 80 g/m² (Nautilus® Classic, Mondi AG, Austria) was used for various tests. Preliminary experiments showed influences of paper direction and paper side used on the later determined fractured surface. To reduce variability of results in the open time, the paper strips were always glued to the glue film with the same direction and side. In step 3, additional paper strips were glued on the glue film every minute, if longer open times were expected. For shorter open times, especially when using higher MDF board and glue temperatures

Fig. 1 Schematic procedure for measuring the open time of PVAc glues



in combination with a low relative humidity, every half minute was tested. Each paper strip was pressed for 6 min before being manually peeled off (step 4). The visual evaluation of the fractured surface was examined after all strips were peeled off. As soon as no larger fragments of the paper remained on the glue film, the open time (time between glue application and assembling of the joint) was considered to be exceeded (step 5). Compared to the standard EN 16556, the proposed method does not measure strength properties of the joint, but only whether the surface of the adhesive still contains enough moisture to wet the paper sufficiently. For each

experimental testing point in Table 2, five repetitions were performed to determine the open time.

2.5 Statistical Analyses

From all 5 repetitions, the arithmetic mean and the corresponding standard deviation were calculated. Furthermore, regression models were calculated to identify significant factors influencing the open times depending on the experiments carried out. An F-test with a significance level of 5% was used to select statistically significant variables and their interactions.

3 Results and Discussion

As a first result, it can be stated that the presented method is well suited for measuring the open assembly time of PVAc adhesives. Compared to other methods, reproducible results can be achieved without the need for large amounts of material and time. Furthermore, the method can also be used to investigate the effects of modifications of the adhesive itself on the open time.

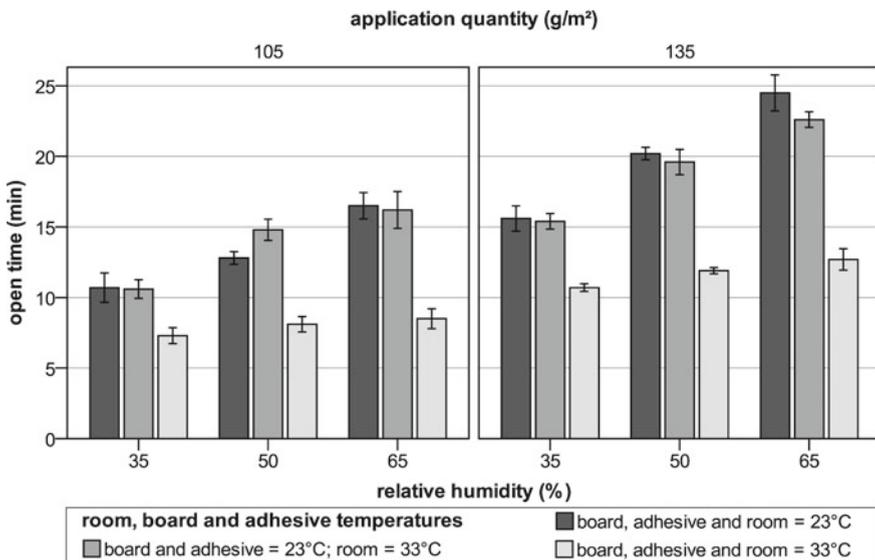


Fig. 2 Open times of D3 PVAc glues at various relative humidities, temperatures and application quantities

The measured open times of the D3 PVAc glue are illustrated in Fig. 2. Each individual bar represents the mean value of five repetitions performed, with the standard deviation displayed as an error bar. A comparison of the two different application quantities shows a clear difference in open time. The more glue is applied, the longer the maximum open time, which is also mentioned in literature (Dunky and Niemz 2002). Interestingly, similar open assembly time values can be achieved with a lower application quantity of 105 g/m^2 at high relative humidity (i.e. 65%) than with a higher application rate of 135 g/m^2 and a simultaneous low relative humidity (i.e. 35%). As a result, the amount of adhesive can be significantly reduced without reducing the open time if air humidity near the adhesive application would be kept high enough. This could lead to distinct savings in glue quantities for PVAc-processing companies, where open assembly time is the limiting factor in the process. However, the relative humidity exhibits a positive influence on the open time for both application quantities. This behaviour is also mentioned by Habenicht (2009). The author further stated that a higher moisture content of the substrate material would cause a prolonged open time, which was not investigated in this study.

The temperatures of the room, MDF board and adhesive influence the open time very differently. Comparing the results in Fig. 2, the measured open time behaves quite similar for each individual relative humidity and PVAc application quantity, when MDF board and glue temperature are always kept constant at $23 \text{ }^\circ\text{C}$. It can be concluded, that there is no considerable change in the open time even if the ambient temperature rises to $33 \text{ }^\circ\text{C}$. However, increasing the MDF board and glue temperature to $33 \text{ }^\circ\text{C}$ leads to a significant reduction of open time as illustrated in the results in Fig. 2. Habenicht (2009) already stated a reduction of the open time with a simultaneous higher temperature of the adherend or ambient air.

Based on the results in Fig. 2, it can be observed that the relative extension of the open time exhibits similar relative changes of open time for the two different glue application quantities. An increase in relative humidity from 35 to 50% seems to be more effective than the increase from 50 to 65% as can be seen by the higher relative changes of the open time. The total relative increase of open time from 35 to 65% relative humidity resulted in prolongations between 47 and 57% for the two glue application quantities and room temperatures investigated. Using a room, MDF board and adhesive temperature of $33 \text{ }^\circ\text{C}$, a considerably smaller relative increase in open time compared to a temperature of $23 \text{ }^\circ\text{C}$ could be observed. Whereas a change in relative humidity from 35 to 65% at $23 \text{ }^\circ\text{C}$ room temperature resulted in a relative increase in open time between 54 and 57%, values of only 16 and 19% could be achieved at a room, MDF board and adhesive temperature of $33 \text{ }^\circ\text{C}$. A possible reason for this could be the lower viscosity due to the increased adhesive temperature as shown in Fig. 3 and hence a faster penetration behaviour.

Figure 4 shows the open times of the investigated D4 PVAc glue at different relative humidities, room temperatures and applied glue quantities. The MDF board and glue temperature were always kept at $23 \text{ }^\circ\text{C}$ to prevent a premature reaction of the D4 PVAc glue. In general, the mean open times are insignificantly lower compared to the results of the D3 PVAc in Fig. 2. A possible reason for this might be the considerably lower viscosity of the D4 PVAc glue as listed in Table 1 and also measured in Fig. 3,