

# Comparative Study of Effect of Addition of Calcium Carbonate and Clay on the Performance Properties of Polyvinyl Acetate Wood Glue

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## Abstract

Polyvinyl alcohol (PVA) stabilized Polyvinyl acetate (PVAc) dispersions-based wood adhesive has poor water and heat resistance. Recently, the addition of fillers in the wood adhesive is one of the most effective ways to enhance the performance of PVAc wood adhesive. Inorganic fillers have unique characteristics to improve the performance of adhesive, such as small size, high surface energy and surface hardness. Hence, the present work investigates the applicability of calcium carbonate and clay incorporated 3% in situ emulsion polymerization PVAc wood adhesive. Effect on physical, thermal and mechanical properties was studied by viscosity, pH, contact angle measurement, differential scanning calorimetry (DSC) and pencil hardness test of films. Emulsions with 3% calcium carbonate and 3% clay were prepared and the shear strength of the applied adhesive on wood was measured. The viscosity of the adhesives was reduced in the case of the addition of calcium carbonate and increased in the case of clay. The mechanical properties like tensile strength of adhesives with calcium carbonate and clay were measured by a universal tensile machine (UTM). Thermal stability was studied by differential scanning calorimetry (DSC). The tensile shear strength demonstrates that clay can improve bonding strength as compared to calcium carbonate of PVAc adhesive in wet conditions. The hardness of PVAc films was also changed positively by the addition of calcium carbonate and clay. Thermal stability of PVAc was significantly improved as calcium carbonate and clay were added to PVAc. Here, we did a comparative study of the effect of the addition of calcium carbonate and clay filler materials in situ polymerization of PVAc on their different properties.

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## Keywords

Polyvinyl Acetate, Clay, Calcium Carbonate, Polyvinyl Alcohol, Wood Adhesive

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## 1. Introduction

Wood adhesives account for a considerable part of the costs and dependency on petroleum sources. In order to decrease the costs, the wood industry tries to use lower cost adhesives, sometimes compromising the quality of the products [1]-[6]. Wood adhesives and wood products are important to the manufacturing of products for construction and furniture production [7]. Adhesives are also used to produce wood composed products, such as plywood, medium density fiberboard (MDF) and oriented strand board (OSB) [8] [9].

Polyvinyl acetate (PVAc) emulsion wood adhesives are mainly used in the non-structural bonding of interior or exterior furniture and other timber industries [10]. PVAc emulsions have certain advantages such as adhesion to a wide variety of surfaces, high molecular weight, high solid content, rapid setting speed, easy modification of water and solvent resistance, good machining characteristics, resistance to micro-organism, resistance to oxidation and ultra violet radiation. PVAc emulsion also has certain disadvantages such as poor performance at elevated temperatures and humid conditions as a wood adhesive. Some research projects have been targeted at modifications of PVAc in order to improve its performance [11] [12] [13].

The modifications can be divided into three main groups: 1) Copolymerizing vinyl acetate with more hydrophobic monomers or functional monomers like acrylic acid, N-Methylol acrylamide [14] [15] [16], 2) blending PVAc with other adhesives or hardeners and the addition of fillers [17] [18] [19] [20] [21]. The performance and application properties of wood adhesives can be regulated by adding several compounds such as tackifiers, adhesion promoters and fillers to adhesives [22] [23] [24] [25] [26].

Fillers are added to emulsion adhesives to reduce cost by replacing resin solid without decreasing total solid, to reduce penetration into porous substrate and to change the rheology of the compound. A selection of the filler depends on 1) Its effect on the rheology. 2) Oil absorption value. The high oil absorption value of fillers raises viscosity markedly, while low oil absorption fillers have less effect on viscosity. Depending on their properties, they can also introduce hardness, stiffness and strength. The following are general purpose fillers that can be added to all polyvinyl acetate homo and copolymers adhesives. They are calcium carbonate, bentonite, calcium silicate, clay mica, nut shell flours, silica, talc, uncooked starches and wood flour [27]. Fillers are in the form of powders, which are added to the emulsion mixture. The choice of good filler material is a function of the binder type and the adhesive type desired. The fillers are playing an

increasingly important role in polymer type applications [28]. Organo-clays derived from natural montmorillonite and modified with quaternary ammonium salt (dimethyl benzyl hydrogenated tallow ammonium) have been shown to improve the properties of polyvinyl acetate [29] and urea-formaldehyde adhesives [30] [31] [32] [33]. To our best knowledge, no research has examined the comparative study between them and the effects of adding calcium carbonate and clay on the bond strength of PVAc at wet conditions so far.

This research is aimed at assessing the bond strength of adhesives with calcium carbonate and clay at dry and wet conditions and finding approaches to improve the bond strength. The shear strength of the specimens was examined to evaluate the water resistance, by which the specimens were immersed in cold water for 24 hr. The water resistance of the cured film was also studied by using a contact angle. Differential Scanning Calorimetry analysis (DSC) was utilized to obtain information on the interaction of fillers with the emulsion and the cured films. DSC result showed a substantial increase in T<sub>g</sub>, with the addition of clay in the in-situ polymerization of VAc based emulsion adhesive which is also confirmed in film hardness testing. The performance properties like tensile shear strength in the dry and wet condition of this clay encapsulated PVAc based wood adhesive was improved. The newly synthesized economic adhesive with improved properties, especially bond strength in wet and dry conditions through the application of calcium carbonate and clay.

## 2. Experimental

### 2.1. Materials

Partially hydrolyzed polyvinyl alcohol (PH PVA) (CAS No. 9002-89-5, % degree of hydrolysis 87% - 89%, Molecular weight 99,000 - 105,000 g/mol) was obtained from Kuraray Cooperative Limited, India. Vinyl acetate (VAc, CAS No. 108-05-4), sodium lauryl sulfate (SLS, CAS No. 68585-34-2), sodium bicarbonate (SBC, CAS No. 144-55-8), potassium persulfate (PPS, CAS No. 7727-21-1), 2-ethyl hexanol (2 EH, CAS No. 104-76-7) and Isothiazolinones preservative (CAS No. 1003-07-2) were acquired from Sigma Aldrich. Calcium carbonate (CaCO<sub>3</sub>, CAS No. 471-34-1, % Moisture content of 1% and Oil absorption value of 19.5 - 20.5 gm/100gm) clay (CAS NO. 1332-58-7, % Moisture content of 1% and Oil absorption value of 37 - 45 gm/100gm) was purchased from 20 Microns Limited. Deionized water (DIW) was used which was prepared and tested in the lab.

### 2.2. Preparation Method

Polymer emulsions were synthesized in a flat-bottom glass reactor equipped with a condenser, stirrer, temperature indicator, metric pump and glass funnel for the addition of additives. Semi-continuous emulsion polymerization process was adopted with continuous supplying thermal energy using a water heating bath. **Table 1** shows the polymer emulsion recipes.

**Table 1.** Composition of the emulsions.

Components	PVAC Homo	PVAc Homo CC	PVAc Homo CL
	Weight % (%)	Weight % (%)	Weight % (%)
Vinyl acetate monomer	46.60	43.60	43.60
Clay	0.00	0.00	3.00
Calcium carbonate	0.00	3.00	0.00
DIW	45.11	45.11	45.11
PH PVA	2.63	2.63	2.63
SBC	0.11	0.11	0.11
SLS	0.01	0.01	0.01
2-Ethyl hexanol	0.01	0.01	0.01
DIW	1.53	1.53	1.53
PSS	0.06	0.06	0.06
DIW	1.20	1.20	1.20
PPS	0.04	0.04	0.04
DIW	2.30	2.30	2.30
Sodium benzoate	0.10	0.10	0.10
DIW	0.15	0.15	0.15
Isothiazolinones Preservative	0.15	0.15	0.15
Total	100	100	100

### 2.3. Colloid Solution Preparation

PVA was first put into a glass reactor. The water was added to them, and the mixture was kept for continuous stirring at 175 rpm. The temperature was slowly raised up to 94°C - 96°C and the final temperature was kept at 94°C - 96°C. The cooking was carried out for 2.5 hr. Then, the solution was cooled down to 25°C and added isothiazolinones preservative. It is used in PVAc Homo.

### 2.4. PVA-Filler Solution Preparation

Fillers like Calcium carbonate in PVAc Homo CC and clay in PVAc Homo CL and PVA were first put into a glass reactor. The water was added to them, and the mixture was kept for continuous stirring at 175 rpm. The temperature was slowly raised up to 94°C - 96°C and the final temperature was kept at 94°C - 96°C. The cooking was carried out for 2.5 hr. Then, the solution was cooled down to 25°C and added isothiazolinones preservative.

### 2.5 PVA Stabilized Emulsion (PVAc Homo)

PH PVA solution was used as a protective colloid for the stabilization of the

emulsion. Sodium lauryl sulphate was used as an anionic surfactant. Sodium bicarbonate is used as buffer. Initiator solution was prepared by dissolving potassium persulfate in deionised water. 2 ethyl hexanol was used to control foam generation in batch. Polymerization was carried out by controlled addition of vinyl acetate (VAM) monomer. After the complete addition of monomer, potassium persulfate solution in water was added and temperature was maintained at 90°C for 1 hr. Then, the emulsion was cooled down to 25°C and added Isothiazolinones preservative.

## 2.6. PVA and Starch Stabilized Emulsion (PVAc Homo CC and PVAc Homo CL)

PH PVA + filler solution was used as a protective colloid for stabilization of emulsion. Sodium lauryl sulphate was used as an anionic surfactant. Sodium bicarbonate is used as buffer. Initiator solution was prepared by dissolving potassium persulfate in Deionised water. 2 ethyl hexanol defoamer was used to control foam generation in batch. Polymerization was carried out by controlled addition of monomer VAM. After the complete addition of monomer, potassium persulfate solution in water was added and temperature was maintained at 90°C for 1 hr. Then, the emulsion was cooled down to 25°C and added isothiazolinones preservative.

## 3. Characterization and Testing

### 3.1. Solid Content

Solids content of emulsion was checked by heating sample (around 1 g) at 120°C for 120 minutes in a hot air oven. Measurements were done in triplicate to minimize error. To measure the solid content the following equation was used,

$$\text{Solid content (\%)} = (W_3 - W_2) / (W_2 - W_1) \times 100\%$$

$W_1$  = Weight of empty weighing pan.

$W_2$  = Weight of Pan with sample before heating.

$W_3$  = Weight of pan with residue after drying till constant weight.

### 3.2. Viscosity and pH

A Brookfield Viscometer RVT was used for calculating the viscosities of the samples. The values were taken at 20 rpm and at a temperature of 30°C. pH of the samples was analysed using a digital pH meter, CL 54 + Toshcon Industries, India. To reduce the error, viscosity and pH measurements were taken 5 times.

### 3.3. Differential Scanning Calorimetry (DSC)

A Perkin Elmer instrument Q 100 DSC has been used for estimating the glass transition temperature ( $T_g$ ) of the samples. During the measurement, an oxygen-free nitrogen stream of 40 mL/min was maintained through the cell. Sample film casted on Teflon was dried at 105°C for 90 min. 5 mg film was analysed directly on DSC by cyclic heating-cool-heat method. The DSC analyses were done

within a range of  $-10^{\circ}\text{C}$  to  $200^{\circ}\text{C}$  at a heating rate of  $10^{\circ}\text{C}\cdot\text{min}^{-1}$ .

### 3.4. Drying Time on Glass Plate

Using an applicator, 200-micron films were casted over a glass plate and the time for drying was measured using a stopwatch. Time taken for full drying of films is noted at  $30^{\circ}\text{C}$ .

### 3.5. Pencil Hardness Test

The pencil hardness test was done using a QHQ-A portable pencil hardness tester, having lead pencils of varying hardness numbers. Using an applicator, 200-micron films were casted over a glass plate. After the films are dried at  $28^{\circ}\text{C}$  for 1 day, the test was conducted following ASTM D 3363 standard.

### 3.6. Water Contact Angle Measurement

The water contact angle was measured using a Rame-Hart Goniometer, Germany at  $28^{\circ}\text{C}$  and 60% relative humidity. To measure the contact angle, the samples are cast over glass substrates with an applicator of 100 micrometers. The test was conducted 5 times to reduce the error. Contact angle measurement gives an idea of the behaviour of prepared samples with respect to water. Moreover, variations in the hydrophobicity in formulations can be analysed using contact angle measurements.

### 3.7. Tensile Shear Strength-Wet and Dry

Shear strength was evaluated at different intervals in joints prepared by gluing beech specimens in a normal climate. Bonded assemblies were tested after keeping them at  $23^{\circ}\text{C}$  and 60% relative humidity after 4 hr, 6 hr, 24 hr and 7 days. In this case, strength was calculated in MPa. Wet strength tests were performed according to condition D3-3 of EN 205 after 7 days in a normal climate, specimens were soaked in water for an additional 24 hr and then tested water resistance.

## 4. Results and Discussions

### 4.1. Solid Content

Solids content of emulsion was checked by heating the sample (around 1 g) at  $120^{\circ}\text{C}$  for 120 min in oven. It was calculated that the solid for both the samples are approximately the same (**Table 2**).

### 4.2. Viscosity and pH

Viscosity and pH of the prepared samples are shown in **Table 2**. Presence of calcium carbonate in PVAc Homo CC sample enhances the pH and makes the system less acidic in nature as compared to PVAc Homo CL. Compared to the PVAc Homo CC, increase in viscosity was observed in PVAc Homo CL containing clay-PVA as a protective colloid. High oil absorption value and smaller particle size of clay resulted in increased in viscosity as compare to PVAc Homo CC.

**Table 2.** Various test results.

Sr. No	Properties	PVAc Homo	PVAc Homo CC	PVAc Homo CL
1	% Solid	46.4	46.4	46.7
2	Viscosity (Poise)	400	320	500
3	pH	4.9	6.55	5.13
4	Drying time on glass plate (% relative humidity-60% Temperature-30°C)	50	70	62
5	Film Hardness	HB	2H	3H
6	Contact angle (°)	45.7 ± 1.39	43.2 ± 2.38	54.3 ± 2.92
7	Glass transition temperature (°C)	106.10	111.18	118.89
	Tensile share strength (Dry) (MPa)			
	4 hr	1.86 ± 0.4	2.08 ± 0.4	2.09 ± 0.4
8	6 hr	3.27 ± 0.4	4.32 ± 0.4	4.17 ± 0.4
	24 hr	4.56 ± 0.4	6.08 ± 0.4	5.70 ± 0.4
	7 Days	6.20 ± 0.4	6.23 ± 0.4	6.23 ± 0.4
9	Tensile shear strength (Wet) (MPa)	1.15 ± 0.3	1.10 ± 0.3	1.43 ± 0.3
	After 24 hr			

### 4.3. Differential Scanning Calorimetry (DSC)

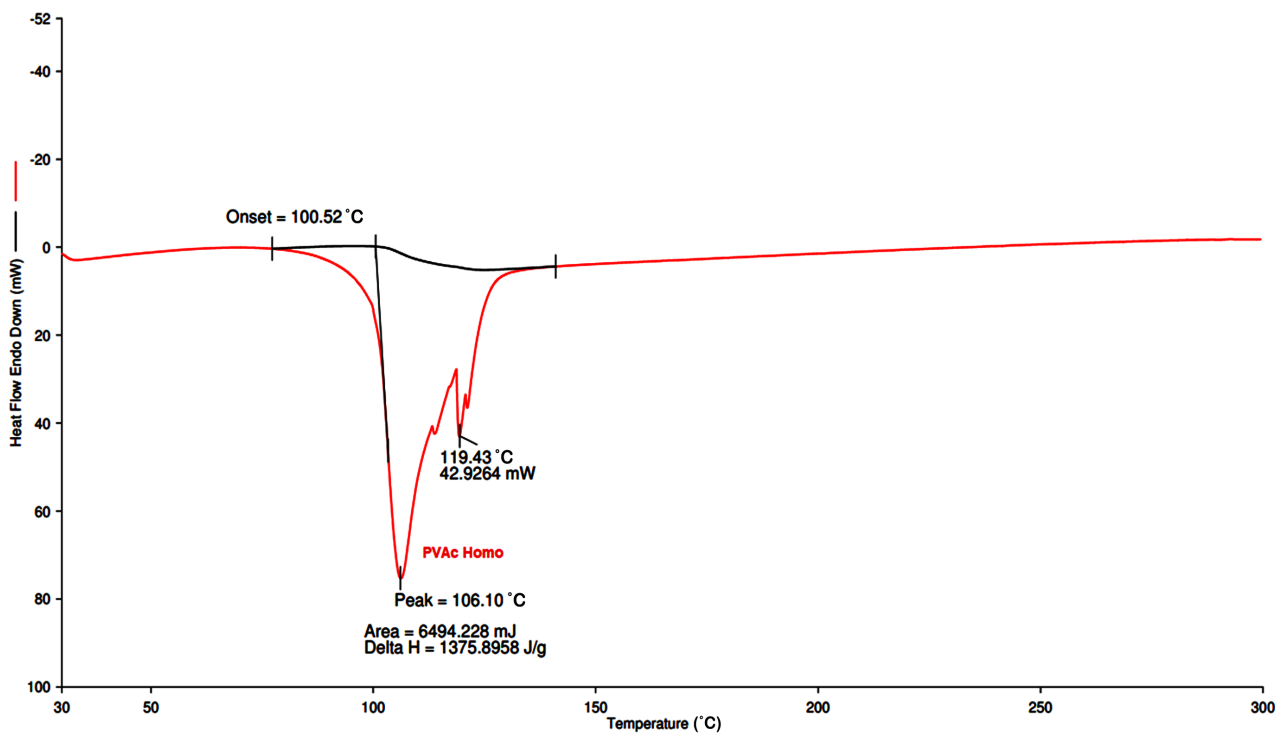
DSC thermograms quantify the thermal stability of samples. The overlaid DSC thermograms for PVAc homo and PVAc Homo CC, PVAc Homo and PVAc Homo CL are shown in **Figure 1**. Both the samples were disintegrated at about their  $T_g$ , mainly attributed to the decompositions of polymer and homopolymer chains. Due to secondary forces in PVAc Homo CL led to the increment of  $T_g$  as compared to PVAc homo CC, due to the complex network of hydrogen bonding formation. The same is observed from the viscosity analysis.

### 4.4. Drying Time on Glass Plate

Drying time test was conducted on glass plate at controlled conditions (50% relative humidity, temperature of 29°C - 31°C). From the result shown in **Table 2**, the drying time for PVAc Homo CC was more than that of PVAc Homo and PVAc Homo CL. The acidity of the filler influenced the drying time on glass plate as shown in **Table 2**.

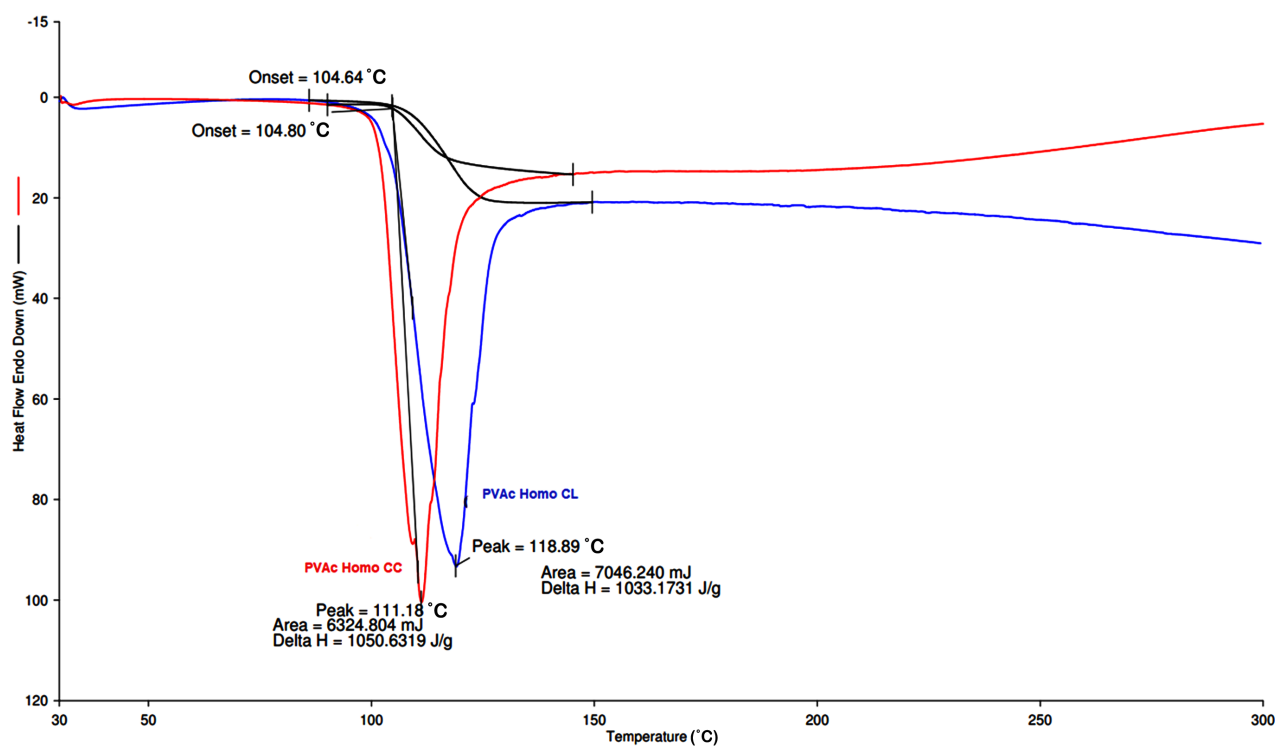
### 4.5. Pencil Hardness of Film

Pencil hardness of polymeric film evaluates the mechanical property. As the addition of clay in PVAc Homo CL, hardness of film was increased as compared to



1) Heat from 30.00 °C to 300.00 °C at 10.00 °C/min

(a)



1) Heat from 30.00 °C to 300.00 °C at 10.00 °C/min

(b)

**Figure 1.** DSC thermograms for (a) PVAc Homo (b) PVAc Homo CC and PVAc Homo CL.



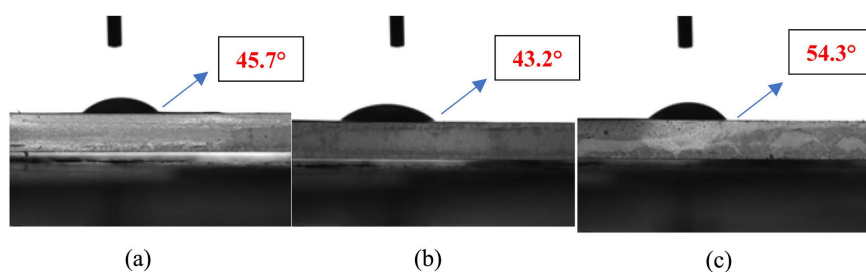
PVAc Homo and PVAc Homo CC as shown in **Table 2**. Clay has high acidic nature hence adhesive formulated from them have quick film formation and faster drying when compared with calcium carbonate. Due to faster drying, slip from sharp pencil due to platelet like structure increased, hence the PVAc Homo CL sample showed a higher value of pencil hardness.

#### 4.6. Water Contact Angle

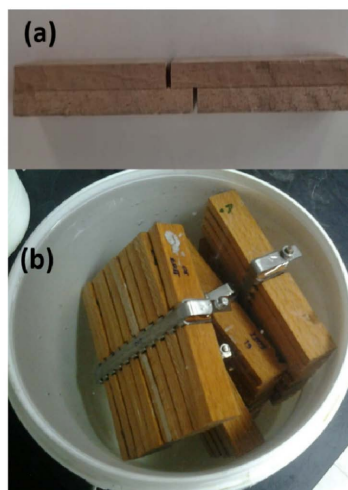
In general water contact angle correlates with the hydrophobic or hydrophilic nature of the samples. Moreover, to correlate with the water resistance properties of PVAc Homo CC and PVAc Homo CL with the PVAc Homo, water contact angle measurement was studied. Distilled water is made in contact with the emulsion samples casted over glass slides and contact angle values were noted. The contact angle of the sample with water is shown in **Figure 2** and the values are labelled in **Table 2**. It is observed that the contact angle value is higher for PVAc Homo CL as compared to PVAc Homo and PVAc Homo CC. Clay has high acidic nature hence emulsion synthesis from them has quick film formation and faster drying when compared with calcium carbonate. Slight reduction in strength of bonded wood specimen in wet condition in PVAc Homo and PVAc Homo CC provides evidence for the same observation.

#### 4.7. Tensile Shear Strength Testing-Wet and Dry

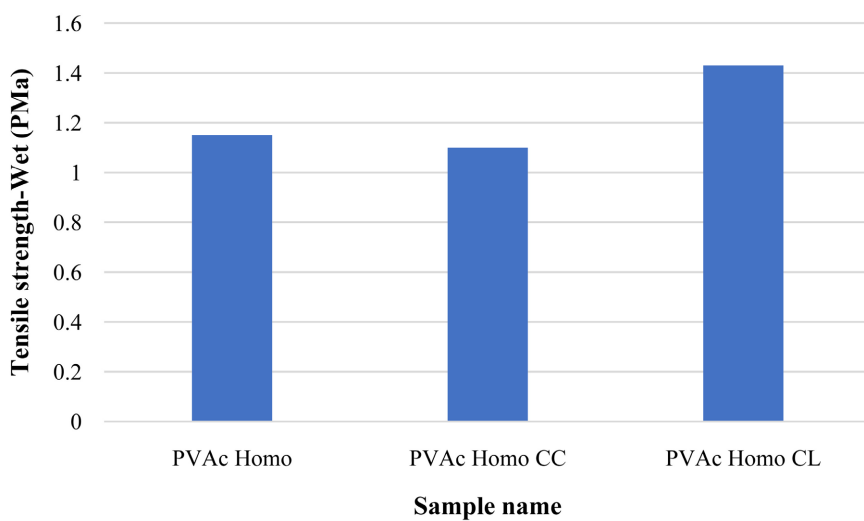
The bonding strength of adhesives was tested on the substrates in dry condition and another set of samples in wet conditions as per EN 204 for water resistance, represented in **Figure 3**. From **Table 2**, it was observed that by the addition of calcium carbonate in PVAc Homo CC, water resistance was slightly decreased as compared to PVAc Homo CL. After 24 hr immersion in water, PVAc Homo CC has lower strength as compared to PVAc Homo CL as shown in **Figure 4**. This is due to platelet like structure of clay particle which act as a barrier for water or moisture through adhesive which was validated in contact angle testing. With the addition of clay and calcium carbonate, dry strength increased in PVAc Homo CC and PVAc Homo CL as compared to PVAc Homo this was due to hardness introduced by calcium carbonate and clay which resulted in faster setting of bond on wood substrate. Handling strength was more due to the high hardness of PVAc Homo CL as shown in **Figure 5**.



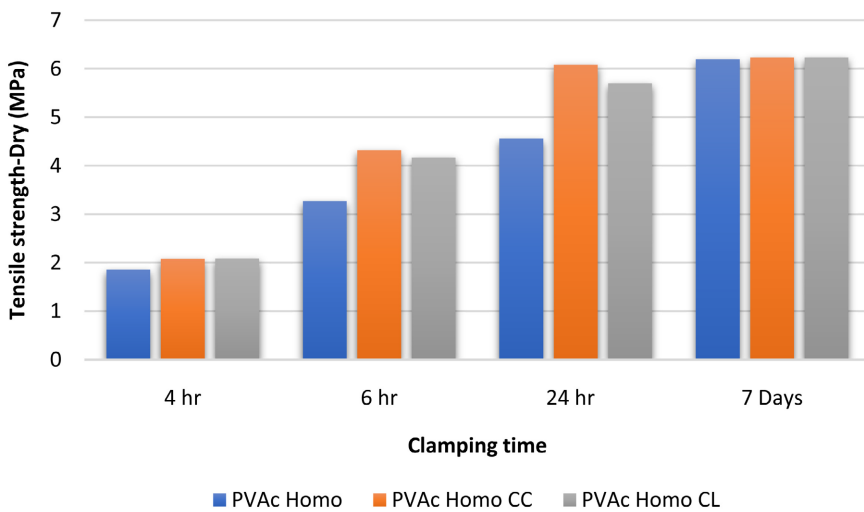
**Figure 2.** Water contact angle images for (a) PVAc Homo (b) PVAc Homo CC (c) PVAc Homo CL.



**Figure 3.** Adhesive bonded wood assembly (a) Dry condition (b) Wet condition.



**Figure 4.** Tensile shear strength in wet conditions.



**Figure 5.** Tensile shear strength in dry condition.

## 5. Conclusion

The calcium carbonate and clay added to in situ emulsion polymerization of polyvinyl acetate (PVAc) improved the thermal properties of adhesives as compared to virgin PVAc Homo, increased entropy factor and activation energy of decomposition of the PVAc. In addition, PVAc with the clay affected other physical features, increasing the viscosity and reducing the pH of PVAc Homo CL. the drying time for PVAc Homo CC was more than that of PVAc Homo and PVAc Homo CL this is due to the acidity of the filler influenced the drying time. Calcium carbonate and clay added in PVAc Homo CC and PVAc Homo CL samples improved the setting speed of dry state shear strength of the bonded wood substrate. The highest values of shear strength were obtained when clay was added *in situ* polymerization of PVAc under wet conditions which were validated in contact angle analysis. The addition of clay increased shear strength in wet conditions by 24% approximately as compared to PVAc Homo and PVAc Homo CC. The newly synthesized economic adhesive has improved properties, especially bond strength in wet and dry conditions through the application of calcium carbonate and clay.

## Conflicts of Interest

The author declares no conflicts of interest regarding the publication of this paper.

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