

Abstract

Tomasiewicz, Ryan. The Adhesion of Paperboard to the Gypsum Core of Wallboard: An Investigation of Adhesive Bond Quality in Response to Paper Production Variables and Relative Humidity. (Under the direction of Dr. Martin A. Hubbe, Dr. John A Heitmann, and Dr. Joel J. Pawlak.)

The research investigated the interactions between paperboard and the gypsum core during the manufacturing process of gypsum wallboard. The goal of this research was to understand the paper properties that can affect the quality of the adhesive bond under simulated test conditions. The categories of paper properties tested include strength properties, surface properties, and paper additives. This research showed that the current test methods in use did not provide enough sensitive and reproducibility data to gain crucial insight into the paper/core adhesive interaction. A new evaluation method must be secured. However, with the results obtained, it was believed that the three types of failure could be described. First, the adhesive bond may fail resulting in a clean peel during bond quality testing. Second, the paper bond network may fail resulting in complete paper coverage of the gypsum core. Third, a mixed failure of adhesive and paper bond resulted in incomplete and varied paper coverage of the gypsum core.

The Adhesion of Paperboard to the Gypsum Core of Wallboard: An Investigation of Adhesive Bond Quality in Response to Paper Production Variables and Relative Humidity.

By

Ryan Tomasiewicz

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Approved by:

Dr. John A. Heitmann

Dr. Joel J. Pawlak

Dr. Martin A. Hubbe
Chair of Committee

Biography

The author was born in Chicago, Illinois on February 15, 1977 to Debra and Raymond Tomasiewicz. He graduated in May 1991 from Hiawatha Elementary School in Berwyn, Illinois. In May of 1995, he graduated with highest honors from Owen-Withee Senior High School. He matriculated to the University of Wisconsin – Stevens Point where he earned a Bachelor of Science degree in Paper Science from the College of Natural Resources in May of 2000. In August of 2000, he began his graduate work on his Master of Science from North Carolina State University in the College of Natural Resources in the Department of Wood and Paper Science. The author's main interests are in wet-end chemistry and properties of paper.

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1 Introduction

1.1 THE IMPORTANCE OF GYPSUM

Hydrous calcium sulphate, gypsum, is a naturally occurring mineral that is composed of calcium sulphate and two molecules of water per repeat unit (di-hydrate). Gypsum has many unique qualities for use in building applications. One important property is that gypsum can be ground into a fine powder, calcined, and, after rehydration, the material can be brought back to its original crystalline composition, corresponding to the rock state, as shown in Figure 1.1.1. The second important property of gypsum is the high water content. This property is critical in creating a low heat transfer coefficient for the material [United States Gypsum 2003], thus making it an excellent fire retardant.

The United States has two main veins of gypsum, which are considered to provide a virtually limitless supply of the material [National Gypsum, 2003]. One vein starts in southwestern Texas and runs to the Niagara River basin in New York State. The other vein begins in the Imperial Valley of California and spreads out into Utah. These gypsum veins are important because gypsum is used in virtually every building application on the planet and is thus important to future growth.



FIGURE 1.1.1 DIHYDRATE GYPSUM CRYSTALS

Thirty-six billion square feet of gypsum wallboard production capacity is available for consumer use in eighty United States plants [National Gypsum, 2003]. The construction boom of the early twenty-first century has helped the wallboard industry giants, such as National Gypsum and United States Gypsum, by increasing product demand. However, the gypsum paper industry will have to make major system upgrades and renovations to meet the demands for gypsum wallboard in the twenty-first century [Balzer and Mohen, 2000].

Because of ISO certification and the ASTM Industrial standards, the physical property demands on wallboard, and thus on gypsum paper liner, make it extremely difficult to manufacture and improve the gypsum product cost effectively. The gypsum industry's trend to lower basis weight of both the paper and the gypsum core poses a particular challenge in meeting tensile, ply-bond, porosity, and smoothness standards set by the various governing bodies [Balzer and Mohen, 2000]. The responsibility for meeting these standards falls upon the gypsum materials scientists, wallboard engineers, and paper scientists. Further understanding of the adhesion of the paperboard to the wallboard composite is essential to future gypsum board production [Balzer and Mohen, 2000].

To understand how the paperboard can affect gypsum wallboard properties, a basic description of current manufacturing techniques is provided. This review contains a brief overview of the manufacturing process, followed by an overview of the patents by major manufacturers that represent the current level of technology used by the wallboard industry. Because the gypsum process involves an aqueous gypsum slurry sandwiched between two sheets of paper, it is also important to discuss the effects of water on paper and the paper itself.

There are two components to wallboard, one being the gypsum core and the second being the paper. In order to understand the adhesive bond quality between the two, it is important to understand the gypsum core. A Master's thesis by Pingel provides a good foundation for work with gypsum and identifies future research applications for paper [Pingel, 1990].

The present thesis builds upon this foundation and addresses possible modifications to current paper production techniques that may improve the adhesive bond and thus the overall physical properties of the gypsum board. This paper begins to uncover the relationship between the paperboard and the gypsum core of wallboard. First, a comprehensive analysis of paper properties that may affect the adhesive bond strength was undertaken. Based on paper property testing, test designed to explore the adhesive bond relationship with moisture were conducted.

2 Literature Review

2.1 CALCIUM SULFATE, THE RAW MATERIAL FOR THE CORE

Calcium Sulfate, CaSO_4 , is a naturally occurring mineral found throughout the world and most commonly called gypsum. This naturally occurring rock is extracted from the earth by mining or quarrying. In North America major veins of gypsum are found in Canada, Mexico, and the United States [Kirk-Othmer, 1992].

TABLE 2.1.1 NOMENCLATURE AND MOLECULAR FORMULA OF GYPSUM.

Common Name	Chemical Name	Molecular Formula
Anhydrite	Anhydrous	CaSO_4
Stucco	Hemihydrate	$\text{CaSO}_4 \cdot 1/2\text{H}_2\text{O}$
Gypsum	Dihydrate	$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$

Naturally, calcium sulfate is found in several forms. Table 2.1.1 shows the common name, abbreviated chemical name, and molecular formula for the three forms of calcium sulfate present during the manufacture of gypsum wallboard. The anhydrous and dihydrate forms of calcium sulfate are commonly found together in the raw, unearthed state. In Table 2.1.2, the physical properties of calcium sulfate are listed for the three forms. The dihydrate when heated to 128°C will transition to the hemihydrate form. The hemihydrate will transition to the anhydrous form at 163°C [Kirk-Othmer, 1992].

It is also important to note that the hemihydrate form of gypsum has two morphological forms, alpha and beta. Although not chemically or mineralogical different, the distinction between the two forms is due to differences in crystal size, surface area, and degree of lattice perfection. The alpha form has well shaped crystal structure of rods and prisms, while the beta form is a needle-like structure, with pores and fine crystals [Anusavice, 1996].

TABLE 2.1.2 PHYSICAL PROPERTIES OF GYPSUM IN ITS THREE FORMS.

Property	Dihydrate	Hemihydrate	Anhydrate
molecular wt.	172.17	145.15	136.14
transition point, °C	128 ^a 163 ^b	163 ^b	
mp ^c , °C	1450	1450	1450
specific gravity	2.32		2.96
solubility at 25°C, g/100 g H ₂ O	0.24	0.30	0.20

^aHemihydrate is formed.

^bAnhydrous material is formed

^cCompound decomposes

In the dihydrate form, calcium sulfate is in a monoclinic crystal form with $a=5.68$, $b=15.18$, $c=6.29$, $\beta = 113^\circ 50'$, (Figure 2.1.1). The structure consists of a calcium ion bonded between two parallel sheets of SO₄ ions. Layers of water molecules separate the double sheets. The calcium ions are coordinated by the oxygen from two molecules of water and from six molecules of SO₄ [Chang, 2002].

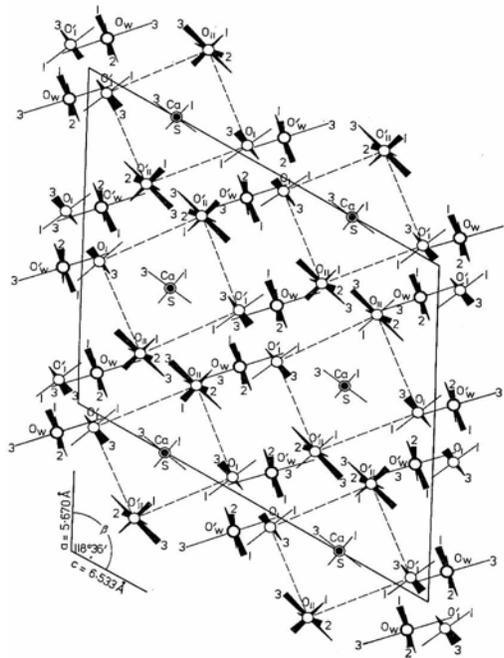
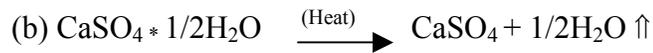


FIGURE 2.1.1 GYPSUM CRYSTAL STRUCTURE [COLE, 1974].

The removal of the water molecules from the gypsum is completed in a batch or continuous kiln. The chemical equations for dehydration of gypsum are shown:



Formula (a) shows the dehydration from the dihydrate form to the hemihydrate, and (b) shows the dehydration from the hemihydrate to the anhydrous form. The approximate heat of dehydration from the dihydrate to the anhydrous form is 108,600 J/mol. The heat of dehydration for the hemihydrate depends on the morphological form alpha or beta being formed from the dihydrate form (84,600 J/mole, 86,700 J/mole, respectively) [Chang, 2002].

The calcination process occurs in several stages. Gypsum begins to lose waters of hydration between 42°C and 49°C. The hemihydrate releases vapor at 116 to 120°C. Once the vapor release has occurred, the hemihydrate can be dried between 149 and 166°C and becomes stucco [Chang, 2002]. Calcination can occur by a batch process or a continuous process.

2.2 GYPSUM BOARD PRODUCTION

The first stage of gypsum wallboard production is the harvesting of gypsum rock from the mine. After Gypsum has been mined, the rock is transported by ship, rail, or truck to the gypsum production plants until needed. From rock pile storage, the gypsum rock is then metered into a large rock dryer where surface water is removed. The rocks are ground by Raymond mills where they are turned into “land plaster”. This land plaster is then calcined to remove approximately three quarters of the water [National Gypsum, 2003]. The dry fine powder is now in a form called stucco.

The basic process of converting stucco into the core material for gypsum board begins with the blending of the stucco with water to create the wallboard slurry in a device known as a pin mixer. In order to control the conversion of slurry to crystalline rock state, various ingredients are added to the slurry mixture such as: accelerators, retarders, starch, *etc.* [National Gypsum, 2003]. The integration of water with the stucco begins the exothermic process of crystallization back to its original rock state.

After mixing, the slurry is transported to a conveyor, where it is spread over a bottom sheet of paper. Then, the top paper merges with the line to form a paper tube that is sandwiched to the rectangular shape. As shown in Figure 2.2.1, the top sheet and the bottom sheet fold over each other to create the rectangular edges. The rectangular sandwich is metered down on a rubber belt to create a uniform distribution of core material. The continuous length of board travels down a long conveyor (600-800 ft) to harden over approximately four minutes. The board is cut into sections and prepared for entry into a drying kiln to remove excess water [National Gypsum, 2003].

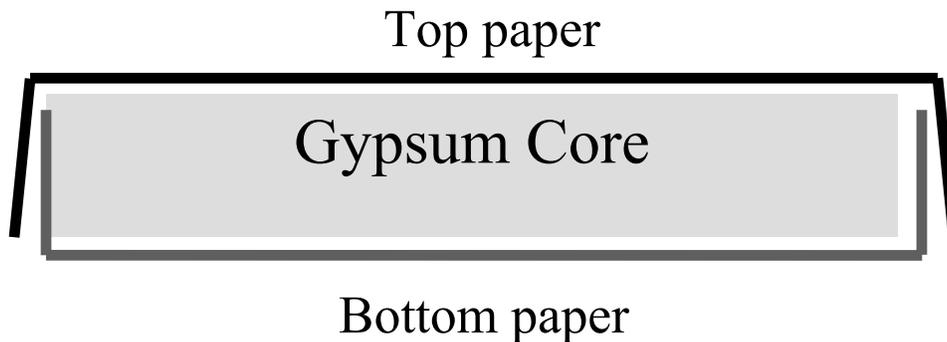


FIGURE 2.2.1 A WALLBOARD SCHEMATIC SHOWING A BOTTOM SHEET WITH GYPSUM SLURRY EXTRUDED ON IT WITH THE SIDE SHEETS COMING TOGETHER AND MERGING WITH THE TOP SHEET TO FORM A RECTANGULAR GYPSUM BOARD.

2.3 GYPSUM WALLBOARD TECHNOLOGY REVIEW

With the basic understanding of wallboard production techniques, Nasman and Wimmerstedt, of the University of Lund/Sweden, in research published in 1993 concluded that the properties of paperboard used in production of the gypsum wallboard are very important, because these properties control the temperature and drying rate of the plaster component [Nasman, 1993]. Lower paperboard density leads to increased permeability of the paper sheet and a higher drying rate of the plaster. However, the paperboard must also provide the gypsum wallboard with tensile strength. In general, as the paperboard density decreases, the tensile strength of the paper sheet decreases. Therefore, one must balance increasing the plaster drying rate with the desired tensile strength of the sheet.

Additionally, Nasman and Wimmerstedt described the transport of soluble species, including salt and starch. It was concluded that starch migration occurred within the core mix concentrating at the paper core interface instead of evenly distributing throughout the core mix. The researchers also determined that starch does not change the drying rates or temperature curves of the product. It was also recommended that the chloride concentration of the slurry must not exceed seventy-five parts per million, in order to prevent paper-core adhesion deterioration [Nasman, 1993].

The application of the gypsum slurry to the paper precedes the drying process. Therefore, it is important to review the previous work to gain an understanding of the penetration of water and how that water plays a role in the development of the adhesive bond. First, it is important to further consider the impact of water penetration on the paper and what technologies are being used to prevent or control penetration.

In 1963, Wurzburg and Mazzarella patented a novel sizing process for paper and paperboard [Wurzburg and Mazzarella, 1963]. The method described the use of substituted dicarboxylic acid anhydrides with hydrophobic groups. The uniqueness of this process was its suitability for use under any pH range used for papermaking acidic, alkaline, or neutral.

This process imparts to the paper a high degree of water resistance against both acidic and alkaline solutions. This agent was typically added at a dosage from one-half percent to five percent of dry paper [Wurzburg and Mazzarella, 1963]. Later in 1974, Wurzburg continued upon the previous patent to include the use of maleic anhydride and an internal olefin to achieve liquid holdout with some additional benefits such as better holdout against acidic liquids (ink, citric acid)[Wurzburg and Mazzarella, 1974].

In a 1968 United States patent, Bieri and Cola discovered that paper covered with an organo-silicone could limit the penetration of water into paper [Bieri, 1968]. This organo-silicone was preferred to be hydrophobic. This chemical could either be a surface treatment or incorporated in the paper structure. This material should be cured in order to establish the cross-linked nature. The product should be added in a range of 0.2 to 2 pounds per ton of paper. Klems and Schull disclosed further related technology in a 1970 Canadian patent. This patent demonstrated that the hydrophobic silicone resin and alum catalyst should be added to achieve a Cobb value of 0.4 to 1.0 grams [Klems, 1970]. The use of silicone is still used in manufacture today for certain grades of gypsum wallboard.

Further developments in 1977, Wain developed a surface treatment for the use in gypsum board production. The treatment involved the use of water-soluble zirconium acetate at a loading of 1 to 2 grams per meter squared. This application occurred immediately before the gypsum board slurry came into contact with the paperboard. This process allowed for a better quality of paper-to-core adhesion [Wain, 1977]. This appears to be the first work cited to indicate the use of chemical bonding to increase adhesive bond quality rather than mechanical entrapment of the gypsum crystals.

Long [1980] developed a method for treating the surface of paperboard starch and stucco. The Gypsum was added at 0.75 to 30% weight, and the starch was added at 5 to 30% weight. The stucco and starch mix was metered onto paperboard in 16 equally spaced strips (per inch). This striped paper pattern allowed for a sufficient area to remain untreated or without starch to achieve a good wet bond with the gypsum core to prevent blow out in the kiln [Long, 1980].

Previous applications of starch were used only in the core formulations of the gypsum wallboard and no coated on the surface to increase adhesive bond quality. The core starch migrated to the gypsum paper interface to protect the gypsum crystal formation, whereas Long placed the starch at the interface.

In 1982, Kennedy patented the use of a substituted succinic acid anhydride [Kennedy, 1982]. Prior art by Wurzburg and Mazzarella [1963] incorporated the substituted succinic acid anhydride at any point during the wet end production of the paper. Kennedy incorporated the substituted succinic acid anhydride as a water-emulsion treated on the surface of the paper. It is important to note that the alkyl succinic acid anhydride (ASA) has been proven to be more effective than the silicone surface size reducing the cost by 30% in previous patented research. This chemical application was applied to achieve a 0.4 to 1.0 gram Cobb value. The solution was coated onto the paper in a 1.15% solids emulsion at 1 pound per ton of 60 pound per thousand square foot of paper [Kennedy, 1982].

2.4 GYPSUM BOARD PAPER PRODUCTION

From the information presented, thus far, one can understand the complex nature of the gypsum core as well as the importance of paper in the gypsum wallboard. The next sections will focus on the paperboard made for the use in gypsum wallboard and the properties of paper important for the use in gypsum wallboard.

Paperboard made for use by the gypsum industry is solely a recycled product. The industry generally uses various waste papers including old-corrugated cardboard, Kraft clippings, and newsprint. This waste paper is pulped, screened and cleaned to later form into paperboard typically on traditional cylinder machines. The pulp can be divided into two color groups depending on the finishing application, such as a gray back or brown Kraft color. The recycled pulp is generally not deinked or bleached.

Generally, the pulp is formed into paperboard consisting of a several layer product with a face side, often called crème face or gray back, and a backside. The crème face is generally gray or crème in color for a finishing affect, while the backside is the traditional Kraft recycled color that will interface with the gypsum core.

Two critical paperboard properties are tensile strength and water absorbency. The tensile strength of the paper provides the overall wallboard with the flexural rigidity and tensile strength. Water absorbency is a critical factor because the gypsum is formed in aqueous slurry that is piped onto the paper. The paper must be able to accept some water to create a wet bond with the gypsum core that is necessary to prevent peel off during the drying phase.

A review of patent literature provides further understanding of gypsum paperboard properties currently used in the industry. Johnstone filed for several patents throughout the 1980's that showed current manufacturing techniques to create paperboard for the production of gypsum wallboard. The main fiber source has been waste paper stock that is controlled to a neutral pH, with a Canadian Standard Freeness of 300 to 550 ml. The filler content was 10 to 35 percent calcium carbonate. The paper additives consisted of an internal sizing agent and surface treatments to prevent the migration of water into the sheet of paper during wallboard production. The internal size was an alkyl-substituted succinic acid anhydride (ASA), and the surface treatments could have been a poly vinyl alcohol, heat-cured silicone, or a paraffin wax emulsion. Also, cationic starch was added to the paper [Johnstone, 1984].

Johnstone found that a neutral sized paperboard had several advantages when making gypsum liner. First, the sheet strength was greater than that of paper made with rosin-alum sizing. Therefore, a weaker fiber source could be utilized. Johnstone found that these fibers under neutral sizing conditions were more conformable during production and made a sheet with greater strength and bonding ability with the gypsum core. Additionally, it was important to create a paper with a great enough porosity to allow for appropriate moisture transfer during the drying phase a gypsum board making [Johnstone, 1982]. The combination of a stronger paper with a good porosity paper made an excellent paperboard for making gypsum wallboard.

2.5 PAPERBOARD AND MOISTURE

The ability of water or water vapor to move through the sheet is essential to paper and gypsum production. In a later section of this paper, the importance of wallboard moisture transport is discussed; therefore, it is essential to understand how paper is affected by moisture. Three aspects to vapor moisture / paper interactions will be discussed in this section. These aspects are equilibrium moisture content in paper, hygro-expansivity of fibers, and diffusion of moisture through paper.

Equilibrium Moisture Content:

Paper is a hygroscopic material, which means water is easily absorbed into fibers. The amount of water that can be absorbed by the fiber at various relative humidities depends on two factors: (a) previous chemical and physical treatments, and (b) prior moisture environments. The previous chemical and/or physical treatment may include various pulping conditions, refining, wet pressing, and wet end additives. The effect of prior moisture environments on paper exhibits a behavior known as hysteresis [Gallay, 1973].

The characteristics of hysteresis behavior are shown in Figure 2.3. As can be seen in the figure, the moisture content of the paper sheet is dependent upon the previous history of the paper sheet. For example, when looking at a specific humidity level (e.g. fifty percent), the amount of water in the fiber during desorption (e.g. seventy-five to fifty percent) is greater than during adsorption (e.g. twenty-five to fifty percent) [Niskanen, 1998].

In addition to paper exhibiting hysteresis behavior, paper fiber may also become hornified. Hornification is important for gypsum papers because of the use of recycled fiber to make the paperboard. Hornification is defined as the irreversible loss of water holding capacity in fibers as the number of humidity cycles increases [Niskanen, 1998].

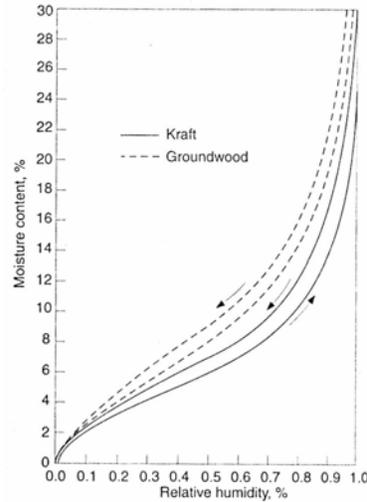


FIGURE 2.5.1 HYSTERSIS CURVE FOR KRAFT AND GROUNDWOOD FIBERS [NISKANEN, 1998].

Two possible explanations of this behavior are hydrogen bond inaccessibility and pore closure. The hydrogen bond inaccessibility theory deals with hydroxyl groups of adjacent microfibrils crosslinking and forming new covalent bonds between microfibrils that are insensitive to water. This makes a portion of the internal surface area of the fibers inaccessible to water and thus reduces the amount of water that may be absorbed by the fibers [Gallay, 1973]. Pore closure reduces the amount of inlets for water to migrate within the fiber structure of individual fibers. Hornification only occurs in chemical pulps [Niskanen, 1998].

The amount of water able to be absorbed or desorbed (hysteresis) from the paper is affected by the fibers' ability to gain and lose water physically (hornification). If a sheet has been recycled a sufficient number of times, it would be likely the paper would not be able to absorb and desorb water as easily as a paper made with virgin pulp. Additionally, the equilibrium moisture contents of the paper would be different.

Hygro-Exspansivity of Fibers and Paper:

Different moisture contents within fibers alter the properties of the paper. When water is absorbed into or out of wood fibers, the fibers will swell, depending on the conditions. Given that fibers are anisotropic, fibers do not swell and shrink equally in all directions. Fibers will change in length by about 1%. Conversely, when fibers swell, the cross-directional area changes about twenty percent, with little axial change [Gallay, 1973].

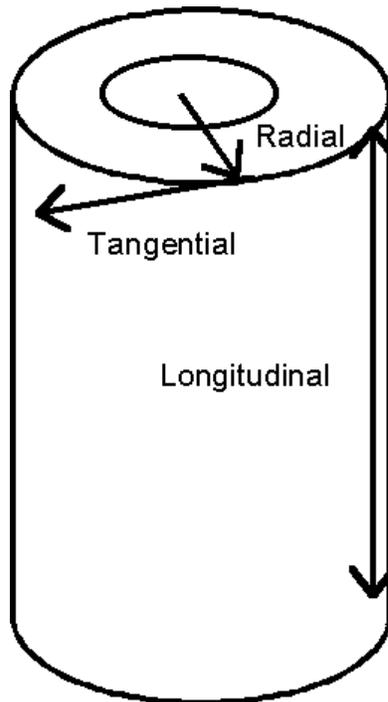


FIGURE 2.5.2 SCHEMATIC OF FIBER SHOWING THE THREE DIRECTIONS.

The origin of anisotropic nature of the swelling can be traced to the fibril angle of the S2 layer. The S2 layer of fibers is largest layer (Figure 2.5.3), especially after chemical pulping and bleaching. The S2 layer fibril angle is approximately ten to thirty degrees relative to the axis of the fiber. This acute angle is approximately parallel to the axis of the fiber. Microfibrils are composed of cellulose chains oriented along the longitudinal axis of the fibril. Along this axis the chain is held together by covalent bonds, which are not sensitive to water.

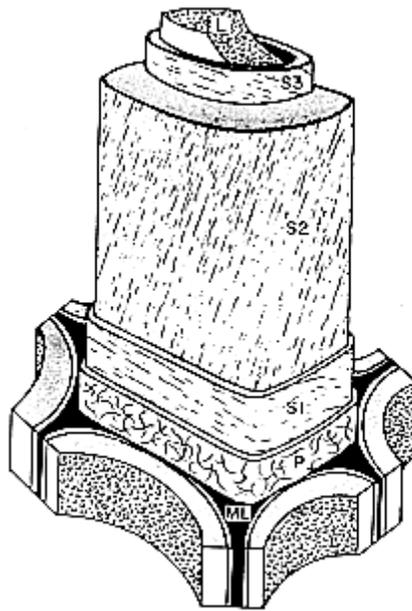


Figure 2.5.3 Cell wall diagram of a fiber noting the layered structure [Smook, 1997].

However, hydrogen bonds hold parallel cellulose chains together. H-bonds are sensitive to water and when exposed to moisture, water may enter the space between the cellulose chains, causing the fibril to expand in the transverse direction. Because of this and the orientation of the fibrils in the axial direction of the fiber, the greatest dimensional changes to the fiber will occur at approximately right angles to the axis of the fiber. This leads to the swelling of the fiber in the tangential and radial directions [Gallay, 1973].

Fiber swelling and shrinkage are affected not only by moisture, but also by the physical and chemical treatment of the pulp. The gypsum industry and most papermakers take advantages of beating when recycling previously dried paper to regain the bonding potential of the fiber and paper strength. An earlier section of this document discussed the chemical nature of swelling; this section will now address physical treatment. The beating or refining fibers is known to release fibrils from the fiber. This will open the fiber to allow for more water to be absorbed.

When water is removed from the paper, the fibrils will lie down on the fibers. This factor is important when considering shrinkage. Drying of paper is an important consideration relative to shrinkage. Machine made papers are dried under tension or restraint [Uesaka, 1994]. The restraint of the paper is caused by the draws from section to section of the paper machine. The cross-direction of the paper is restrained only by the web pressure and not by a tension force as in the machine direction. Therefore, paper will shrink more on a paper machine in the cross direction, resulting in less dried-in strain. In the machine direction, the fibers are prevented from shrinking due to the tension. For further consideration, handsheets can be made with dried-in strain (like machine made papers) or no strain (freely dried). The dried-in strain, or lack of it, is important when considering paper as a layered network of fibers [Uesaka, 1994]. For gypsum papers, the manufacturers will often dry the sheet to very low or to no moisture, then add moisture back into the sheet to the desired level.

The hygro-expansion of multi-layered paper is more complicated than single-ply papers. One must not just consider the expansion of the fibers, but also consider the effect of sheet structure and inter-fiber bonding as well. Uesaka and Qi reached two major conclusions regarding these additional factor effecting papers sheets [Uesaka and Qi, 1994].

The hygro-expansivity of the paper sheet may affect the paper-gypsum core bond. This occurs when the dry paper sheet has moisture applied to it during gypsum board manufacturing. Hill and Ridge [1959] showed that the gypsum core expands by 0.1 percent and shrinks by 0.3 percent change in length during drying. Therefore, amount that the core and the paper shrink is over ten times different. This will lead to dried-in stress at the interface between the gypsum core and the paperboard.

Diffusion of Moisture through Paper:

In gypsum board, assuming that moisture transport in the plane of the board is negligible, moisture must first diffuse through the paperboard before entering the core material or affecting the core/paperboard bond. Therefore, it is important to understand the diffusion of moisture through paper and paperboard. Gypsum board is often subjected to dynamic humidity conditions in application and therefore measurement of the diffusion of water in paperboard under dynamic conditions may aid in explaining the behavior of gypsum board.

Moisture has been described as migrating through paper by five mechanisms: vapor-phase diffusion, Knudsen diffusion, surface diffusion, bulk-solid diffusion, and capillary transport [Liang, 1990]. Vapor phase diffusion is where gas is transported through pores. The vapor phase is dependant on temperature, but independent of relative humidity. Knudsen diffusion also occurs in the gas phase, but only occurs in pores with a diameter less than 100 Angstroms. Knudsen diffusion is dependent on temperature and independent of relative humidity. For vapor-phase and Knudsen diffusion the effect of relative humidity removes the effect of fibers swelling. Surface diffusion is an absorbed diffusion that occurs on the surface of fiber. Bulk-solid diffusion occurs in the absorbed phase, but occurs within the material structure of the fiber. Both surface diffusion and bulk-solid diffusion coefficient increase with relative humidity. Therefore, capillary transport or condensed phase transport can only occur when the pores are filled [Nilsson, Wilhelmsson, Stenstrom, 1993].

From the literature, further work on moisture diffusion is required due to controversy over the dominant method of transport [Radhakrishnan, Chatterjee, & Ramarao, 2000]. This paper focuses on work performed at high humidity levels. Liang et al. found that capillary or condensed-phased transport is an important transport mechanism at high relative humidity levels, while gas-phase transport is a dominant method below 60% relative humidity [Liang, 1990]. Liang et al. indicated that as porosity increases, at least two different rates of transport change: gas phase transport increases while the condensed-phase transport decreases. The work of Liang et al. contradicted Alhen's [1970] earlier findings that condensed phase transport was important at lower levels of relative humidity.

However, in support of Liang et al., Nilsson et al. [1993] found that gas-phase transport was a dominant mechanism and was independent of relative humidity. Nilsson, et al. used relative humidity values less than 58% in the present research. For the general body of knowledge, this work adds to the confusion in developing a concrete theory of moisture transport. Additionally, the present research uses humidities at levels above 58% relative humidity.

To continue Nilsson work, Radhakrishnan, Chatterjee, and Ramarao [2000] found that condensed phase transport becomes increasingly important after 60% relative humidity. Figure 2.5.4 shows the results of Radhakrishnan et al and the results of Nilsson et al. This plot shows the moisture diffusion coefficients as a function average relative humidity. At higher relative humidity the diffusion coefficient increased exponentially and agrees with the previous work of Nilsson et al. Based on the work of Liang [1990], Nilsson [1993], and Radhakrishnan [2000], it appears that at higher relative humidity, condensed phased diffusion of water becomes the dominant mechanism over the other four diffusion methods (bulk-gas, bulk-solid, surface, and Knudsen).

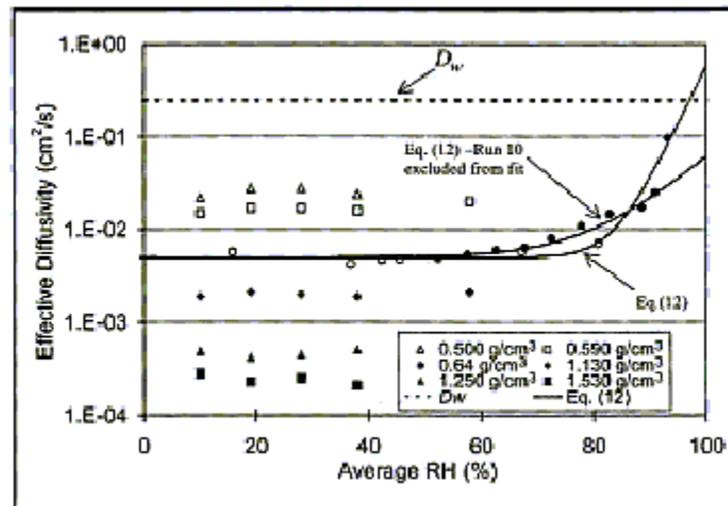


FIGURE 2.5.4 FIGURE TAKEN FROM RADHAKRISHNAN [2000] SHOWING THE EFFECTIVE MOISTURE DIFFUSIVITY IN BLEACHED KRAFT PAPER AS STUDY BY RADHAKRISHNAN [2000] AND NILSSON [1990].

2.6 OUT-OF-PLANE PAPER BEHAVIOR

For gypsum boards, the out of plane behavior is another paper property of interest. Commercial manufacture of paper results in a dried-in strain occurring in the machine direction of the paper. Because fibers are aligned preferentially in the machine direction, the fiber strength as well as the degree of bonding contributes directly to the tensile strength in the machine direction, while fiber bonding predominantly gives paper its strength in the thickness direction (z direction). These two facts explain why the MD tensile strength is two orders of magnitude larger than the other two directions [Lundh and Fellers, 2001]. Common tests used to explore the z-directional strength properties or the z-direction toughness are the Scott Bond tester and the z-directional tensile tester [Koubas, 1995].

The wet strain on paper during manufacture can affect various paper properties, including density and tensile properties for all three directions (MD, CD, & ZD). When a force is applied to a paper sheet in the machine direction during manufacture, the sheet in the MD responds to the strain. This can cause as much as a two factor change in the z-directional stiffness and z-direction strength. The reduction of bonding in the z-direction due to strain and resulting fiber alignment causes the increase in z-directional stiffness and z-directional strengths [Baum, 1984].

Baum, Pers, Shepar, and Ave'Lallemant [1984] conducted several experiments involving wet pressing and the z direction (thickness) of paper. From these experiments, it was determined that wet pressing had a significant effect on the out-of-plane behavior of paper. Experiments showed that a change in the level of wet pressing from 25 psi to 100 psi decreased the anisotropy ratio of R_{xz} from 75 to 55. It was suggested that the ratio would continue to drop further if pressure were increased [Baum, 1984]. Additionally, work by Stratton found that increasing the density by wet pressing could result in as much as a factor of four increase in the out-of-plane elastic stiffness [Stratton, 1993].

Further work by Lundh and Fellers [2001] showed the effects of grammage and refining on z-directional toughness. It was shown that highly beaten pulps had higher z-directional tensile strength than lightly beaten pulps due the amount of bonds being formed as a result of beating. It was also shown that the grammage affects z-directional toughnesses with higher grammage resulting in higher toughness. This was believed to be the result of the increasing three-dimensional nature of the paper as grammage increased.

Also, Lundh and Fellers [2001] looked at the delamination of the paper by creating a delaminating zone in multi-ply sheets by varying the properties in each ply. Lund and Fellers created a three-ply paper with a thin delaminating zone in the middle, surrounded by two thick zones of paper. This experiment showed that the two thick zones were protected, and the weakest zone delaminated. The absence of fiber bridging between the sheets of paper caused this delaminating zone.

Of the several experiments performed by Lundh and Fellers [2001], a small moisture study is especially important to the present research. This small set of experiments showed that as moisture was increased for three different types of paper (paperboard, sack, and liner board), the z-directional toughness decreased.

2.7 PINGEL'S THESIS

Laura J. Pingel conducted master's research related to bonding in paper-gypsum composite structures. This thesis looked at the paper-gypsum interface by visual, spectroscopic, thermal, x-ray, and mechanical techniques [Pingel, 1990].

From Pingel's six conclusions and experimental work the following items can be drawn that are relevant to this investigation. While testing bond quality by varying the combination of alum, starch, and sizing in the paper, Pingel observed starch was a key core material in the gypsum core composite and theorized that the starch limited the penetration of gypsum core material into the paper, resulting in a stronger bond. Next, in the absence of starch, other additives can be incorporated in the fiber furnish, leading to good bonding.

Two major additives found to produce desired results are coupling agents. Titanate and zirconate were found to improve bonding while a third coupling agent, silane, was found to be ineffective at improving bonding [Pingel, 1990]. Additional experiments were conducted to evaluate chemicals that inhibited core penetration into the paper. These paper additives that limit the penetration of gypsum into the core improved bonding.

When experimenting with various hardwood kraft, softwood kraft and groundwood furnish blends, it was determined that without starch in the core formulation, furnishes with high hardwood and groundwood content had good bond qualities. In the presence of starch in the formulation, all fiber furnishes led to good bonding.

The previous results were concluded based on both porosity and bulk characteristics as well as by physical analysis during testing. The research conducted showed that alum and starch included in the fiber furnish had no discernable effect in the paper [Pingel, 1990].

Additionally, a final set of tests was conducted to determine the levels of penetration by chemical analysis. This information showed the distance of chemical penetration into the board. This information confirmed the porosity and bulk implications of penetration from the fiber furnish blends.

It should be noted that bond quality was given only three rating possibilities (good, good/poor, and poor) and the thesis did not list the numerical values obtained during the ninety-degree peel test. The peel strength test is a measure of force required to create the plane of failure [Pingel, 1990]. The gypsum board is placed on a sled apparatus and the paper is hooked. The paper is pulled from the surface with a constant speed and the amount of maximum force required to pull a strip is measured. The advantage of this system is the accurate measurement of the force required to break the paper to core bond and the ability to maintain the ninety-degree angle during the pull. Also, Pingel's samples were limited to one laboratory gypsum board per condition.

3 Objectives

As is evident from the preceding section, the currently available literature relating to the effect of paper properties on gypsum wallboard production is very limited. Several companies have successfully patented technologies. However, there is very little published research related to the adhesion of the paperboard to the gypsum core. It was proposed to study the following items to begin exploring the deficiencies in the literature:

It is proposed that physical properties of the paper can affect the adhesive strength of the gypsum core bond to the paper. It is believed that the bulk, porosity, and water penetration properties of the paper are three key parameters that can affect adhesive strength.

It is proposed that manipulation of the paper surface in contact with gypsum core can affect the strength of the adhesive bond.

It is proposed that by strategically limiting the penetration of gypsum slurry into the paper, the resulting sizing affect will limit starch migration, resulting in a better quality bond.

It is proposed the surface strength of the paper measured by wax pick can be used as a predictor of bond quality at the time of manufacturing of the paper.

4 Experimental

This section will give an overview of the materials and procedures used in the investigation of the adhesive bond quality. Two sets of machine-made paper samples were received from a major manufacturer for testing of the properties of the paper. Two types of handsheet materials were used. The first set was planned to simulate the actual production process; however the ability to clean the recycled pulp over various trials resulted in the decision to use a model pulp from pre-consumer boxboard. Descriptions of the two types of laboratory board making procedure are described due to a change in process conditions. A description of process terminology is provided. Finally, several sections are devoted to the testing of the properties of paper and the modification of these standard procedures.

4.1 MEASUREMENT OF ADHESIVE BOND QUALITY

The measurement of bond quality has been conducted using at least two techniques. The first method is a peel energy or strength measurement, while the second method is a determination of peel failure location (peel test). The adhesive bond quality for this research was evaluated by using the second method (peel test).

The peel test begins with the conditioning of the samples at either 70°F and 50% relative humidity (70/50) or 90°F and 90 % relative humidity (90/90). The 90°F / 90 % condition may be maintained for either 2 hour, resulting in a *2 hour adhesive bond quality test*, or 20 hours, resulting in a *20 hour adhesive bond quality test*. The 70°F/50% r.h test condition is maintained for at least 24 hours, resulting in the *70/50 adhesive bond quality test*. After the boards were conditioned, the boards were measure with a resistive moisture meter (T-8 meter by Aqua Measure Instrument Company) to ensure consistent moisture pickup. Then, the gypsum boards were scored and the samples were broke along the scored line and the paper was pulled back from the board at a 90-degree angle with as consistent force as possible by hand (Figure 4.1.1).

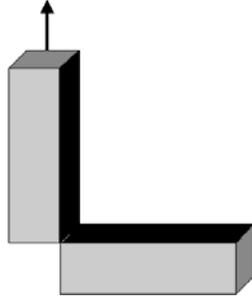


FIGURE 4.1.1 DIAGRAM OF PEEL TEST SHOWING THE PAPER (BLACK AREA) BEING PULLED AT A 90 DEGREE ANGLE AWAY FROM THE CORE MATERIAL (GRAY AREA).

The revealed area was evaluated for how much of the gypsum core was exposed as a percentage of the whole area. A visual observation using a plastic overlay with defined squares is made to measure the amount of squares with exposed core. The value is recorded as a percent peel. Higher values of percent peel are considered as an indication of poor end-use performance. The advantage of this method is the ability to understand the location of the plane of failure, its relationship to the end-use application, and the lack of special specialized equipment. The disadvantage of this method is the variability of the peel angle, force of peel, peel rate, and also the variability associated with different technicians.

4.2 PAPER BOARD TEST SAMPLES – COMMERCIAL

To begin to understand how paper interacts at the paper/gypsum core interface, a battery of physical property tests were performed on the paper to look for readily quantifiable correlations. Eight samples of commercial paperboard were obtained, and each of these samples had been previously evaluated for adhesion strength. Each sample was assigned a letter (A-O) for testing.

4.3 HANDSHEET #1

Old corrugated container (OCC) and newsprint flyleaf fiber materials were received from a commercial recycling source. The two fiber sources were soaked for a minimum of four hours prior to mixing at approximately 2.5% consistency. After being mixed for two hours in a large Hobart mixer to break up into a workable pulp, the fiber was then prepared for forming into handsheets by TAPPI Method T205 om-88. After disintegration the OCC and flyleaf were combined together in the appropriate ratio and diluted according to T205 om-88.

4.4 HANDSHEET #2 AND #3

A bundle of cardboard postal boxes was obtained from a local postal agency in a lot to be used for handsheet #2 and #3. The two fiber sources were soaked for a minimum of four hours prior to mixing at approximately 2.5% consistency. Mixing was carried out for two hours in a large Hobart mixer to break up the material into a workable pulp. The fiber was then formed into handsheets according to TAPPI Method T205 om-88.

4.5 GYPSUM LAB BOARD PRODUCTION

Lab board making is an essential part of the testing process. It is extremely important to develop a constant procedure for making the board. To ensure accuracy, reproducibility, and reliability of the results, a timing scheme of preparation was developed and all gypsum boards were made in duplicate. A step-by-step procedure is given in the following paragraphs.

Lab Board Procedure

First, pre-weigh all materials as listed in Table 4.6.1. Second, at the time boards are to be made, measure out the Ball Mill Accelerator (BMA). Third, add the BMA to the stucco and mix throughout. Place the BMA and stucco mix in the Hobart mixer at medium speed. Fourth, measure out 120°F tap water for both the stucco mix and the foam generation. Start the foam generation and also start a timer. At twenty seconds, run the pulp blender for thirty seconds. At the two-minute mark, record the level of foam that has been generated. At two minutes and twenty seconds, run the pulp blender again for thirty seconds.

TABLE 4.5.1 ORIGINAL MATERIALS LIST USED FOR MAKING GYPSUM BOARD.

Material	Amount
Stucco	870 g
Paper	2.0 g
Pot Ash	0.25 g
Starch	4.0 g
Accelerant	1.8 g
Retarding Agent	1.0 g
Dispersant	2.0 g
Soap	5.0 g
H ₂ O (Core)	710 g
H ₂ O (Foam)	158 g

Upon completion of the thirty seconds, add the retardant and dispersant to the mixer. Begin the preset timer (fifty-five seconds) and add the blender contents into the Hobart mixer containing the gypsum and stucco. Add the generated foam to the Hobart mixer and mix on high speed for ten seconds. Pour the mixer contents into the prepared gypsum envelope and close the forming apparatus.

At three minutes and fifteen seconds, begin to assess the quarter pound set time of the gypsum core and record the results. At the eight minute mark, cut the board to appropriate dimensions and measure its weight. At the eleven minute mark, check the wet bond (ten minute wet bond) of the board by peeling the paper from the core (of the cut off section) and record the relative amount of core area exposed in terms of percent of area covered.

4.6 GYPSUM LAB BOARDING DRYING

Lab board drying was conducted in a forced-air drying oven that could accommodate the four lab boards made during production. The boards are placed into the oven. After drying for eleven minutes, the boards are flipped (right to left) and shifted in the following manner. The board in the bottom position (#4) is placed on the top position (#1). The top position is moved down to the second position (#2) and so on for board number two and three.

At twenty-eight minutes, the boards are rotated ninety degrees on the horizontal and shifted vertically as previously described. At thirty-five minutes, check the moisture reading of the boards. Until the boards read nine percent moisture, continue to dry the boards in the following manner. Place the boards with the highest moisture on the top shelf and continue adding in a descending manner. At seventeen minute intervals, continue the flip and rotation cycle as before.

4.7 FORMATION OF TRIPLE PLY HANDSHEETS

In order to create multi-ply handsheets, individually formed layers were pressed together before wet pressing and drying. As demonstrated in Figure 4.8.1, the sixty pound per thousand square feet handsheet was comprised of three layers: surface, middle, and interface layer.

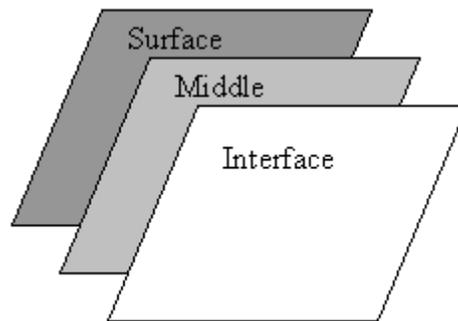


FIGURE 4.7.1 A PICTURE OF THE TERMINOLOGY USED FOR DESCRIBING THE 3-PLY HANDSHEET STRUCTURE USED TO MODEL CYCLINDER BOARD MACHINES.

The forming of the individual layers was performed using the TAPPI Method T 205 om-88 with the following exceptions: Each layer was couched off with application of the least amount of pressure from the brass couch roll. After couching the first sheet, the second sheet was formed and the sheet was couch off onto the previously made sheet until the three-layered product was formed.

After the composite was formed, the combined sheet was placed into a Carver press to be first pressed at 80 psig for three minutes and a second press with new blotters at 80 psig for three minutes. The handsheets were dried in a speed drier at 300°F until bone dry and then allowed to equilibrate in the fifty percent relative humidity environment.

4.8 FORMATION OF SINGLE-PLY HANDSHEETS

The forming of single-ply handsheets was performed using TAPPI method T 205 om-88 with the following exceptions: The basis weight of handsheets was fifty-pound per thousand square feet. After each sheet was formed, it was placed into a Carver press to be first pressed at 80 psig for three minute. The blotter papers were changed and a second press with new blotters was conducted at 80 psig for three minutes. The handsheets were dried until bone dry and then allowed to equilibrate in the fifty percent relative humidity environment.

4.9 PHYSICAL PROPERTY TESTING – TAPPI

Table 4.10.1 indicates the TAPPI methods used for physical testing of paperboard and handsheets.

TABLE 4.9.1 PHYSICAL TEST PROPERTIES AND THE TAPPI METHOD USED FOR TESTING.

Physical Test	TAPPI Method
Caliper	T 411 om-89
Conditioning	T 402 om-88
Contact Angle	T 458 cm-94
Freeness	T 227 om-85
Grammage	T 410 om-88
Gurley	T 460 om-88
Scott Bond	T 569 om-88
Sheffield	T 538 om-88
TAPPI Cobb	T 441 om-84
Tensile	T 494 om-88
Wax Pick	T459 om-88

4.10 PHYSICAL PROPERTY TESTING – MODIFIED COBB

The modified Cobb test was performed by using TAPPI Method T 441 with the exception of the use of 120°F water. This modification was done to better approximate the gypsum board slurry temperature during manufacturing.

4.11 PHYSICAL PROPERTY TESTING – MD AND ANTI-MD DETERMINATION

Before samples were tested for Scott Bond, the paper was subjected to machine direction and anti-machine direction determination. As seen in Figure 4.11.1, the machine direction is the direction in which the paper made down the Fourdriner table toward the dry end while the anti-machine direction would be the in the direction from the wet press section to the headbox, opposite of the papermaking process. To determine the direction of the paper, two half-inch notches were cut one-inch apart into the paper sample. The notches were placed on both end of the paper in the MD dimension.

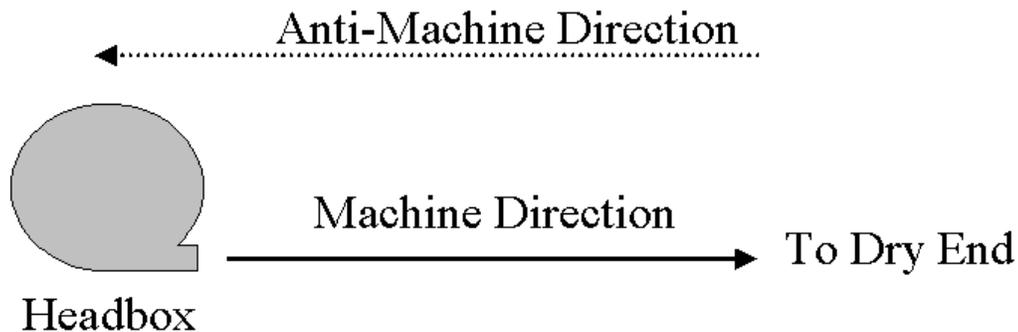


FIGURE 4.11.1 MD AND ANTI-MD SCHEMATIC.

While pressure was applied to each side of the notch, the notch was pulled over the paper at a 90-degree angle until the strip was freed from the paper. After pulling each notch, the longer strip implied that the machine direction is in the direction the strip was made. After this determination was made, samples were prepared following TAPPI Method T 569 om-88.

The samples were placed in the Scott Bond testing apparatus, so that the pendulum swings in the desired direction of testing, swinging with the machine direction or against the machine direction.

4.12 PHYSICAL PROPERTY TESTING – PROFILOMETRY

Profilometry tests were conducted on a Form Talysurf Series 2. The profilometer has a 10 mg stylus load with a vertical resolution of 5 nm and a range of 6 mm. Paper samples were mounted on ceramic tiles, which provided a rigid and relatively straight backing material.

From the profilometry data a number of parameters were calculated. The following briefly describes each of the parameters. It is important to note that all variables are factored into a mean line projection determined by least squares regression to provide a reference plane. R_a is the arithmetic mean of the departure of the profile. R_p is the maximum height of the profile above the mean. R_t is the maximum peak to valley height. R_q is the root mean square of the deviation from the reference line. R_{ku} is the kurtosis, which describes the shape of the amplitude distribution or its sharpness. R_{sk} is the skewness measure of the symmetry of the line of amplitude distortion curve above the mean.

4.13 WIRE IMPRESSION TECHNIQUE

After soaking the paper samples in water for 30 seconds, a wire impression was made on the surface by pressing a wire mesh on to the paper. The wire meshes were pressed into the sheets for 5 minutes at 70 psig. After impression, samples were reconditioned in a standard condition room. The mesh screen were sizes of 18x18, 24x24, and 30x30.

4.14 MOISTURE EXPERIMENTS WITH PAPER AND STARCH

Experiments were designed to understand the rate of moisture pickup and equilibrium moisture content of paperboard, uncooked and cooked starch, and paperboard coated with cooked starch. The samples were conditioned at 70°F/50% relative humidity for a minimum of 24 hours. The samples were then placed in a humidity chamber at 90°F/90% relative humidity. The initial mass of the samples was recorded. The mass of the samples was periodically recorded over the next 48 hours. After samples reached an equilibrium moisture content, they were removed from the chamber and dried at 105°C for 24 hours to determine the oven dried moisture content.

The paperboard samples were prepared according to Table 4.15.1. Contact paper was applied to the described surface in order to create a vapor/liquid barrier in the sheet. Two other samples were tested, cooked starch and an uncooked starch.

TABLE 4.14.1 PAPERBOARD SAMPLE DESIGN SCHEME FOR MOISTURE EXPERIEMENTS.

Sample	Starch Coating	Contact Paper on Core Interface	Contact Paper on Grayback
1	No	No	No
2	No	Yes	No
3	No	No	Yes
4	Yes	No	No
5	Yes	Yes	No
6	Yes	No	Yes

5 Results and Discussion

5.1 PROPOSED MODEL OF ADHESIVE BOND AND ADHESIVE BOND FAILURES

The adhesive bond between paper and gypsum board is proposed to be as follows. In Figure 5.1.1, the paper, top (light gray), and the gypsum core, bottom (light gray), are bonded together by an “unknown” interface layer, middle (dotted). This “unknown” layer is believed to be predominantly composed of paper, gypsum crystals, and starch. The strength, S , of the respective material (P for paper, I for interface; C for core) in the z -direction may be used to characterize the adhesive bond quality. Keeping in mind that adhesive bond quality is an indication of failure location, one can see four cases that exist during failure.

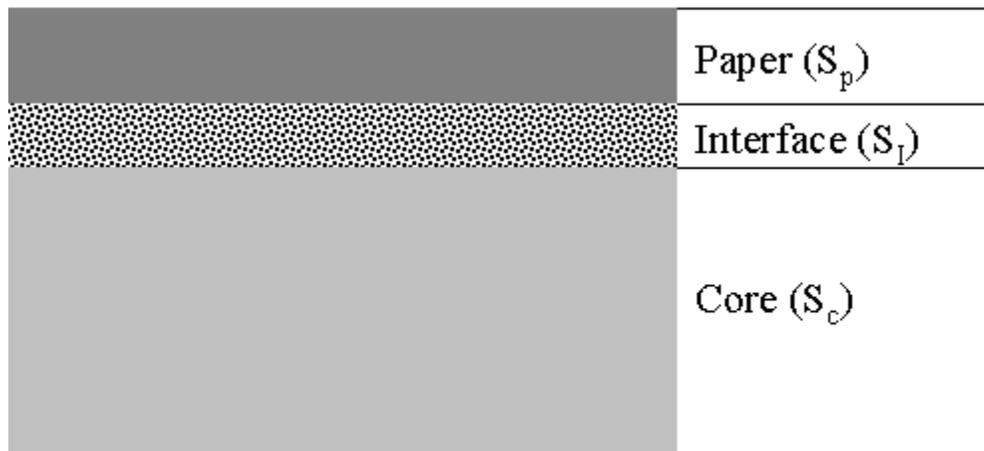


FIGURE 5.1.1 PROPOSED DIAGRAM OF PAPER TO GYPSUM CORE BOND.

Case I:

Case I is a mode of failure occurring within the paper sheet as seen in Figure 5.1.2. This fracture means that the strength of the paper bond (S_P) is less than the strength of the interface bond (S_I) and the strength of the core bond (S_C). This fracture results in a zero percent peel value, which is desirable

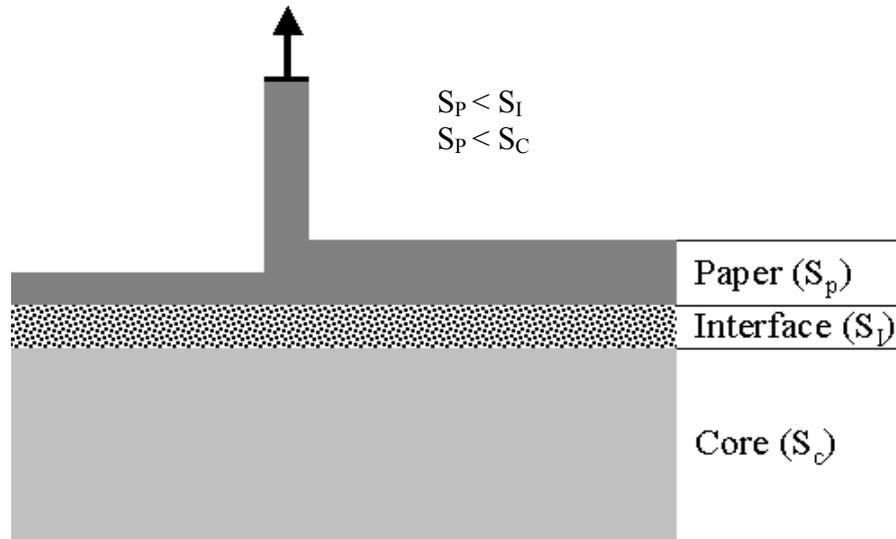


FIGURE 5.1.2 PROPOSED CASE I - FAILURE WITHIN THE PAPER STRUCTURE.

Case II:

Case II is a mode of failure occurring within the gypsum core as seen in Figure 5.1.3. This failure means that the strength of the core bond (S_c) is less than the strength of the interface bond (S_i) and the strength of the paper bond (S_p). This fracture results in a 100 percent peel test, which is undesirable.

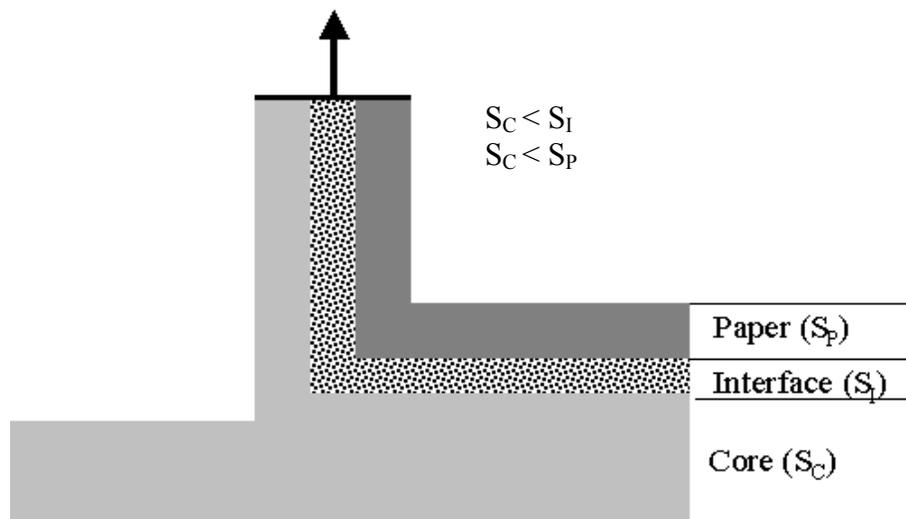


FIGURE 5.1.3 PROPOSED CASE II - FAILURE WITHIN THE GYPSUM CORE.

Case III:

Case III is a mode failure occurring within the “unknown” interface layer as seen in Figure 5.1.4. This fracture means that the strength of the interface bond (S_I) is less than strength of the paper bond (S_P) the and the strength of the core bond (S_C). There is no percent peel value associated with this failure, however it is possible that visual inspections could discern the difference between a complete a core failure and an interface failure.

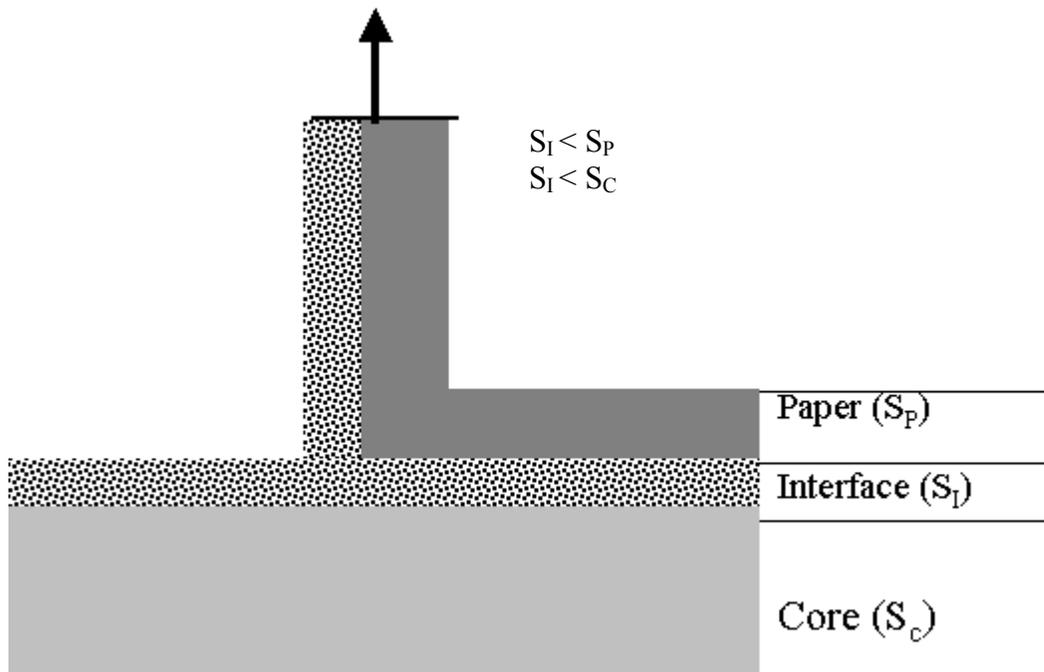


FIGURE 5.1.4 PROPOSED CASE III - FAILURE WITHIN THE "UNKNOWN" INTERFACE.

Case IV:

Case IV has a mixed mode were case I must be occurring because paper remains on the surface, however a combination of cases II and III may also be occurring. This fracture results in peel test value between 0 and 100 percent, but not equal to either.

From the model described above, one sees that some information may be discerned about the strength of each of the principle components in gypsum board. One also sees that the adhesive bond quality is related to the relative strength of the material rather than the absolute strength. It is important to keep the relative strengths in mind when interrupting adhesive bond.

5.2 ADHESIVE BOND QUALITY DATA ANALYSIS

The initial point chosen to begin the investigation was to examine the relationship between the three adhesive bond quality tests. The adhesive bond data results (Table 5.2.1) that were received with paperboard samples were evaluated. Figure 5.2.1 shows the graph of 70°F/50% r.h. conditioned results versus the 2 hours at 90°F/90% r.h. bond results and 20 hours at 90°F/90% r.h. bond results. The 20 hours humidified bond, identified by the squares (red), correlated to the 70°F/50% bond fairly well with the 20 hour humidified bond, while the 2 hours humidified bond, identified by the diamonds (blue), showed little correlation. The 2 hours humidified bond was not capable of differentiating samples below a 5 percent 70°F/50% r.h. bond quality value. Figure 5.2.2 shows the comparison of the 2 hours humidified bond versus the 20 hours humidified bond. There appeared to be no correlation. The data showed little differentiation of the data at below 60 percent humidified bond for the 20 hours humidified bond. At values higher than 80 percent humidified bond, the data were scattered.

In light of the correlations between the 70°F/50% r.h. bond quality and the 20 hours at 90°F/90% r.h. bond quality, the lack of correlation between the 70°F/50% r.h. bond quality and the 2 hours at 90°F/90% r.h. bond quality is somewhat surprising. However, one may find an explanation in the fact that a dynamic condition of moisture absorption exists for the 2 hours at 90°F/90% r.h.. As seen in Figure 5.2.3, adhesive bond qualities of samples A through C have been plotted against the three time conditions. All three samples (A-C) begin and end at approximately the sample position. However, during the 2 hour @ 90°F/90% r.h. bond condition the samples undergo a shift due to the moisture addition. The cause of this shift is the dynamic moisture condition at the 2-hour time, as shown later in section 5.8.

TABLE 5.2.1 ADHESIVE BOND QUALITY RESULTS FOR THE THREE HUMIDIFIED CONDITIONS

Sample	Bond Quality Data		
		2 Hour @	20 Hour @
	70°F / 50 % R.H. (% peel)	90°F / 90 % R.H. (% peel)	90°F / 90 % R.H. (% peel)
A	0	36	5
B	1	52	2
C	0	79	5
D	7	86	20
E	11	88	50
F	7	90	29
G	9	98	66
H	29	99	86

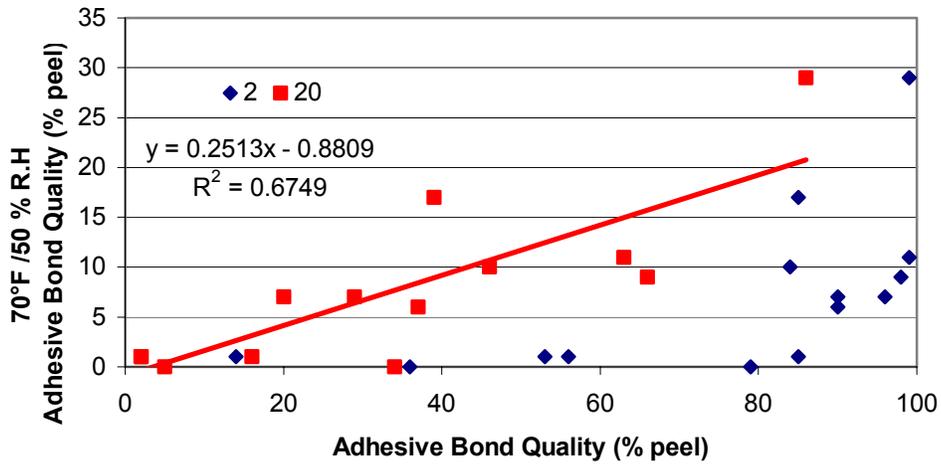


FIGURE 5.2.1 A COMPARISON OF 2 HOUR, DIAMONDS, AND 20 HOUR (90°F/90% R.H.), SQUARES, ADHESIVE BOND QUALITY RESULTS WITH 70°F/50% R.H. ADHESIVE BOND QUALITY RESULTS.

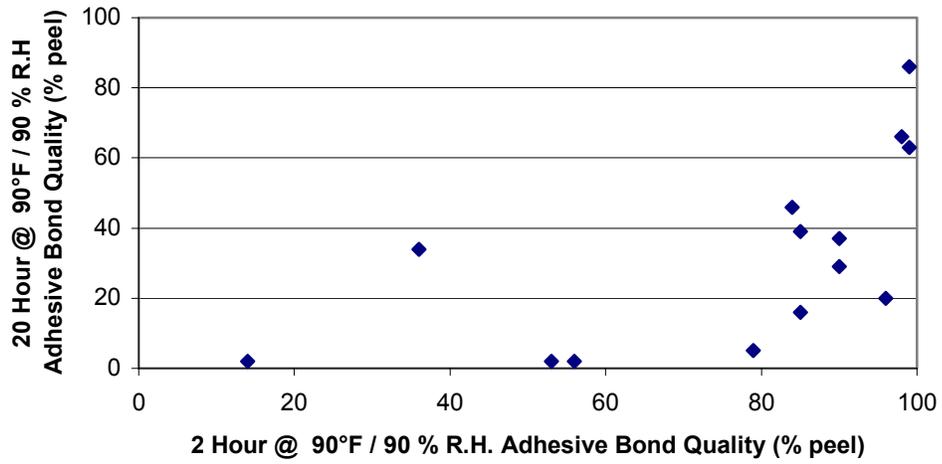


FIGURE 5.2.2 A COMPARISON OF 2 HOURS AND 20 HOURS (90°F/90% R.H.) HUMIDIFIED ADHESIVE BOND QUALITY RESULTS.

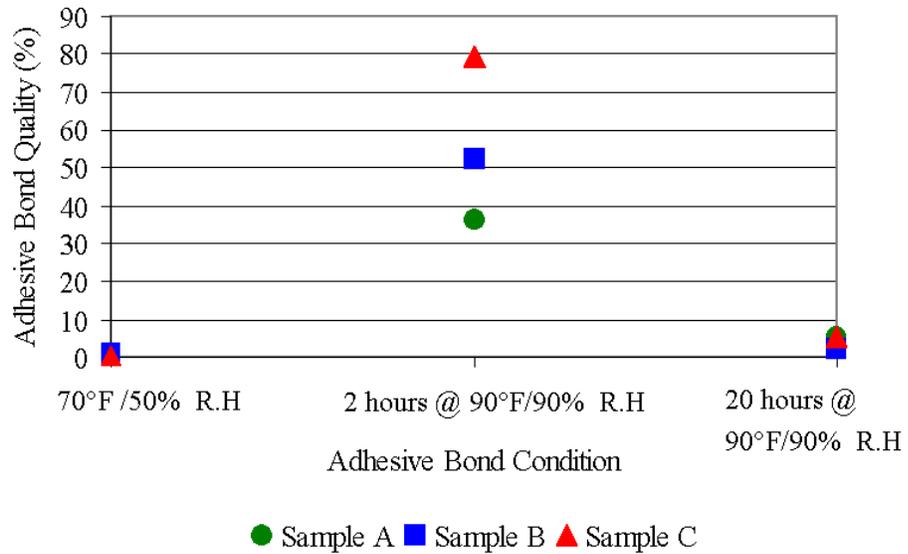


FIGURE 5.2.3 CHANGE IN ADHESIVE BOND QUALITY WITH TIME.

5.3 BASIC PROPERTIES OF PAPER SAMPLES

A thorough analysis of paper properties was undertaken to determine what paper property or paper properties affect adhesive bond quality. Table 5.3.1 contains the basic physical test properties, including basis weight, caliper, apparent density and bulk. The basis weight and caliper showed no correlation with any of the adhesive bond quality data results. However, a generalization was observed; the basis weights of better adhesive bonding paper or preferred modes of failure (case I) were generally below 46 (lb/1000 ft²), while the poorer adhesive bonding paper or not preferred mode of failure (case IV) were generally higher than 49 (lb/1000 ft²) in basis weight.

Samples A-C had a mean apparent density of approximately 44.5 with a range of approximately 3. The 70°F/50% and the 20 hours at 90°F/90% r.h. adhesive bond results average 0.33% and 4%, respectively for samples A-C. However, when the density increased in samples D-H, there was a significant increase in both the 70°F/50% and the 20 hours at 90°F/90% r.h. adhesive bond results (12.6 and 50.2, respectively). Therefore, there appears to be a relationship between the density and the 70°F/50% and the 20 hours at 90°F/90% r.h. adhesive bond results.

Table 5.3.2 shows the results of surface property testing TAPPI Cobb, modified Cobb, contact angle, Gurley porosity, and Sheffield smoothness. The contact angle, Gurley porosity, and Sheffield smoothness showed no relationship to the any of the adhesive bond quality data. Neither the TAPPI Cobb nor the modified Cobb tests showed a relationship with the bond data. As expected with increased temperature, the modified Cobb showed consistently higher water absorptions than the TAPPI standard method. In light of the previous work by Pingel [1990], the paper properties of porosity, the Cobb tests, and potentially the contact angle would have been expected to show a significant relationship. However, Pingel [1990] focused on limiting the penetration of slurry into the paper and did not focus on paper properties, but on the physical penetration of the gypsum slurry into the paper to prove that limiting the penetration of the slurry was essential.

TABLE 5.3.1 RESULTS OF BASE PAPER PROPERTIES COMPARED TO ADHESIVE BOND QUALITY RESULTS.

Sample	Bond Quality Data			Basis Weight (lb/1000 ft ²)	Apparent		
	70°F / 50 % R.H.	2 Hour @ 90°F / 90 % R.H	20 Hour @ 90°F / 90 % R.H		Caliper (mils)	Density (lb/ft ³)	Bulk (ft ³ /lb)
	(% peel)	(% peel)	(% peel)				
A	0	36	5	44.03	12.75	46.78	0.021
B	1	52	2	45.07	11.85	44.51	0.022
C	0	79	5	45.51	11.45	43.42	0.023
D	7	86	20	43.77	13.85	50.52	0.020
E	11	88	50	51.48	12.75	54.70	0.018
F	7	90	29	49.32	14.25	58.57	0.017
G	9	98	66	50.03	13.60	56.70	0.018
H	29	99	86	49.99	13.48	56.16	0.018

TABLE 5.3.2 RESULTS OF SURFACE PROPERTIES COMPARED TO ADHESIVE BOND QUALITY RESULTS.

Sample	Bond Quality Data			TAPPI Modified Contact			Gurley	Sheffield
	70°F / 50 % R.H.	2 Hour @ 90°F / 90 % R.H	20 Hour @ 90°F / 90 % R.H	Cobb	Cobb	Angle	Porosity	Smoothness
	(% peel)	(% peel)	(% peel)	(g/m ²)	(g/m ²)	(degree)	(sec)	(sheff.)
A	0	36	5	26	50	73.6	142	339
B	1	52	2	26	48	70.2	100	314
C	0	79	5	27	48	69.0	128	313
D	7	86	20	33	58	83.8	59	362
E	11	88	50	25	46	76.2	123	320
F	7	90	29	26	49	72.0	106	313
G	9	98	66	25	46	78.5	99	285
H	29	99	86	28	46	75.5	105	316

Table 5.3.3 contains the data collected from Fiber Quality Analysis by soaking and gentle disintegration of the sheet. Using the various length measurements, there was no significant trend in the data, nor did the level of fines percentage indicate a trend. Table 5.3.4 contains the data collected from scraping the core-interface side of the paper. The results do not have an affected range of values (0.837 to 0.972 mm) sufficient to show a significant change in arithmetic mean; however the slightly greater value of arithmetic mean combined with profilometry discussed later may indicate a combined correlation.

Table 5.3.5 displays the results of physical strength testing for the eight samples. The first parameter of breaking length had some possible correlations. The strongest correlation for the breaking length variables was the MD breaking length versus 70°F/50% r.h bond test graphed in Figure 5.3.1. The linear trend line has a R² value of 0.7309, which accounts for approximately 85 percent of the variability in the data.

TABLE 5.3.3 RESULTS OF FIBER QUALITY ANALYSIS COMPARED TO ADHESIVE BOND QUALITY RESULTS.

Sample	Bond Quality			Mean Length			% Fines	
	2 Hour @		20 Hour @	Arithmetic (mm)	Lw (mm)	Ww (mm)	Arithmetic (%)	Lw (%)
	70°F / 50 % R.H. (% peel)	90°F / 90 % R.H. (% peel)	90°F / 90 % R.H (% peel)					
A	0	36	5	0.746	1.246	1.942	51.52	14.19
B	1	53	2	0.674	1.078	1.624	57.32	18.66
C	0	79	5	0.628	1.006	1.648	60.56	21.87
D	11	88	50	0.748	1.154	1.647	57.13	16.75
E	7	90	29	0.774	1.351	2.124	55.42	15.17
F	7	96	20	0.707	1.242	2.183	59.77	19.05
G	9	98	66	0.629	1.017	1.635	61.09	22.39
H	29	99	86	0.612	0.944	1.379	56.46	19.78

TABLE 5.3.4 RESULTS OF FIBER QUALITY ANALYSIS - CORE SCRAPPING COMPARED TO ADHESIVE BOND QUALITY.

Sample	Bond Quality			Mean Length			% Fines	
	2 Hour @		20 Hour @	Arithmetic	Lw	Ww	Arithmetic	Lw
	70°F / 50 %	90°F / 90 %	90°F / 90 %					
	R.H.	% R.H	R.H	(mm)	(mm)	(mm)	(%)	(%)
(% peel)	(% peel)	(% peel)						
A	0	36	5	0.913	1.548	2.377	46.24	9.71
B	1	53	2	0.972	1.639	2.427	49.17	10.12
C	0	79	5	0.97	1.667	2.489	49.57	10.41
D	11	88	50	0.967	1.683	2.509	51.27	10.91
E	7	90	29	0.935	1.58	2.439	49.23	10.59
F	7	96	20	0.896	1.447	2.121	48.63	10.77
G	9	98	66	0.873	1.524	2.431	46.09	10.14
H	29	99	86	0.837	1.396	2.051	51.93	12.94

TABLE 5.3.5 RESULTS OF PHYSICAL STRENGTH PROPERTIES COMPARED TO ADHESIVE BOND QUALITY.

Sample	Bond Quality Data			MD	CD Breaking	MD/CD	Scott Bond	
	2 Hour @		20 Hour @				Breaking	Length
	70°F / 50 %	90°F / 90 %	90°F / 90 %	Length	Length	Ratio	ft-lb	ft-lb
	% R.H.	R.H	R.H	(km)	(km)			
(% peel)	(% peel)	(% peel)						
A	0	36	5	4.75	2.08	2.29	46.40	47.00
B	1	52	2	5.37	1.46	3.68	80.80	83.00
C	0	79	5	4.99	2.03	2.46	84.80	97.80
D	7	86	20	5.77	1.79	3.21	88.80	91.20
E	11	88	50	5.49	1.58	3.48	65.20	82.80
F	7	90	29	5.67	1.59	3.57	66.60	70.60
G	9	98	66	5.52	1.57	3.51	54.20	60.00
H	26	99	86	6.20	1.74	3.57	79.80	80.20

Figures 5.3.2 and 5.3.3 graph the MD breaking length versus the 2 hours and 20 hours at 90°F/90% r.h. bond test data, respectively. These data result in R^2 values of 0.5627 and 0.5379, representing approximately seventy-three to seventy-five percent of the variability in the data collected. The CD breaking length and the MD/CD ratio of breaking lengths did not correlate well to the any of the three conditions. Additionally, the Scott Bond results (Figure5.35) did not correlate to any of the three bond conditions. However, the machine direction samples always had a higher Scott Bond than the anti-machine direction samples. Overall, these data indicated that a paper with low in-plane strength would perform better for gypsum wallboard. Relating this conclusion back to the model of adhesion, the paper strength (S_p) would be manufactured lower than the relative strength of the interface (S_i) and the strength of the gypsum board (S_c). Additionally, Scott bon results do not support creating a gypsum paperboard of either a high or low z-direction toughness.

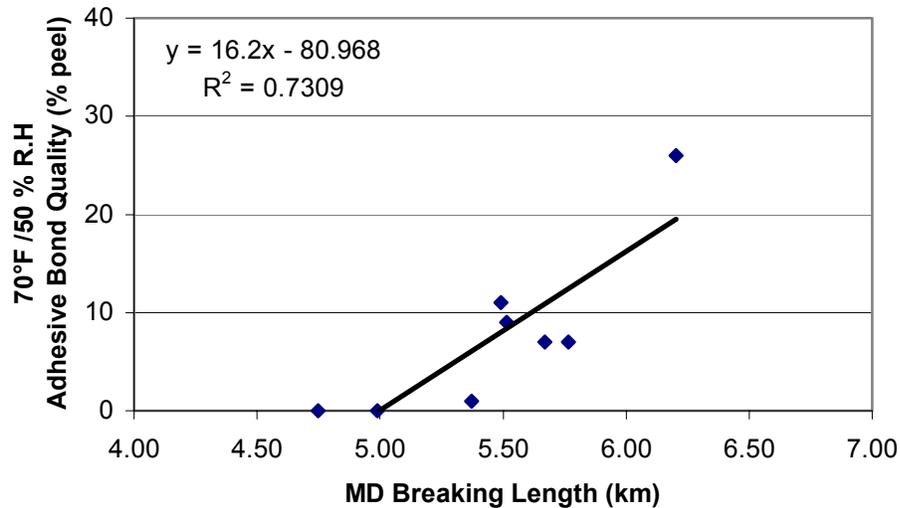


FIGURE 5.3.1 A COMPARISON OF MD BREAKING LENGTH AND THE RESULTS OF ADHESIVE BOND QUALITY AFTER CONDITIONING IN 70°F / 50% R.H.

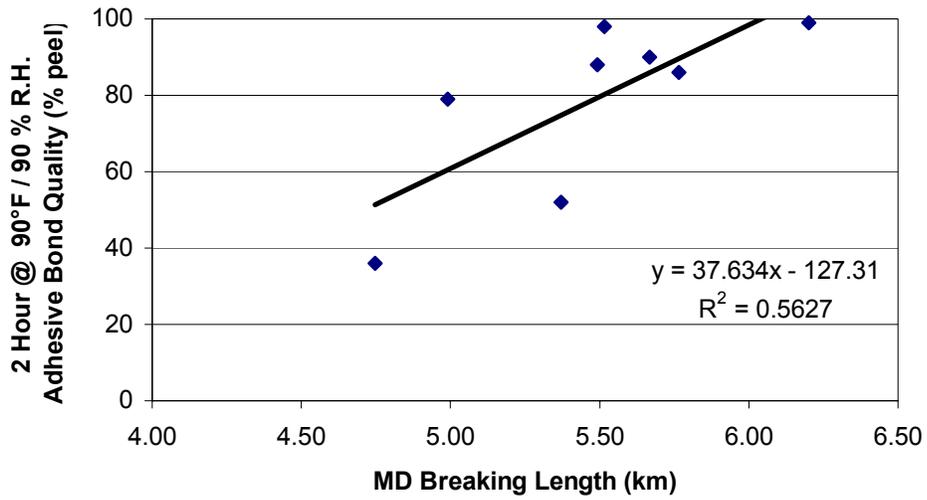


FIGURE 5.3.2 A COMPARISON OF MD BREAKING LENGTH AND THE RESULTS OF ADHESIVE BOND QUALITY AFTER 2 HOUR @ 90°F / 90 % R.H.

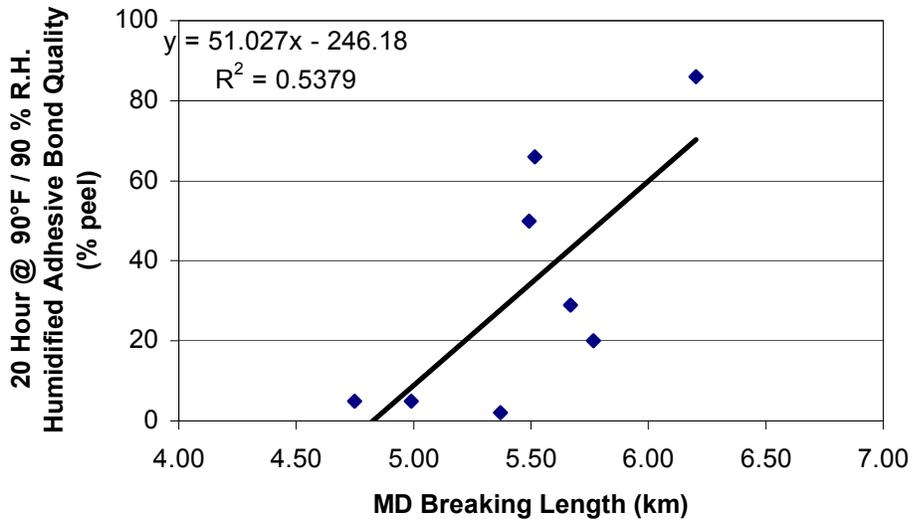


FIGURE 5.3.3 A COMPARISON OF MD BREAKING LENGTH AND THE RESULTS OF ADHESIVE BOND QUALITY AFTER 20 HOUR @ 90°F / 90 % R.H.

TABLE 5.3.6 RESULTS OF PROFILOMETRY COMPARED TO THE ADHESIVE BOND QUALITY.

Sample	Bond Quality Data								
	2 Hour @		20 Hour @						
	70°F / 50	90°F / 90 %	90°F / 90 %						
	% R.H.	R.H	R.H						
(% peel)	(% peel)	(% peel)	R _a MD	R _a CD	R _p MD	R _p CD	R _t MD	R _t CD	
A	0	36	5	6.48	10.61	18.07	23.85	54.25	36.34
B	1	52	2	7.83	8.02	19.30	21.10	38.97	21.10
C	0	79	5	na	na	na	na	na	na
D	7	86	20	6.49	8.43	14.87	18.76	35.14	47.35
E	11	88	50	7.71	9.33	17.24	17.68	61.09	44.21
F	7	90	29	5.22	7.62	11.73	15.16	29.47	13.84
G	9	98	66	4.41	7.37	11.79	17.99	25.84	41.15
H	29	99	86	11.87	6.03	24.49	16.52	56.74	34.09

TABLE 5.3.7 CONTINUED.

Sample	Bond Quality Data								
	2 Hour @		20 Hour @						
	70°F / 50	90°F / 90	90°F / 90 %						
	% R.H.	% R.H	R.H						
(% peel)	(% peel)	(% peel)	R _q MD	R _q CD	R _{ku} MD	R _{ku} CD	R _{ku} MD	R _{sk} CD	
A	0	36	5	7.99	12.95	2.52	2.52	0.14	-0.37
B	1	52	2	9.68	9.56	2.11	2.46	-0.12	0.00
C	0	79	5	na	na	na	na	na	na
D	7	86	20	7.97	8.25	2.60	2.86	-0.34	-0.42
E	11	88	50	9.25	9.25	2.53	2.20	-0.19	-0.32
F	7	90	29	17.04	9.20	2.56	2.45	-0.36	-0.50
G	9	98	66	5.45	9.19	2.57	2.74	-0.05	-0.14
H	29	99	86	13.76	7.47	2.14	2.69	-0.27	-0.08

Profilometry measurements were conducted on the seven board samples provided. The only parameter to show a slight possible relationship was Ra, the mean variation in height. This could show that height variation affected the ability for the gypsum to bond with the paper. Such an effect might be thought to occur either by a physical connection or a chemical consequence of the additional surface area that is created. In comparison to other physical tests, the Ra value should be similar to the smoothness test. These other physical test parameters mentioned did not result in high correlation, meaning the apparent slight correlation between mean variation in height and adhesive bond quality may be fortuitous. Additionally, because no strong correlations existed among the seven tested samples for profilometry, it was decided not to run a test on sample C.

5.4 PAPER SURFACE MANIPULATION

Starch Coating

Having examined a number of mechanical, dimensional, and surface properties of paper and only finding mild relationships between these properties and adhesive bond quality, the focus of the investigation shifted to look at enhancing the strength of the interface (S_I). With the understanding that starch plays a vital role in the formation of a good adhesive bond, two different starch coatings were drawn down on machine-made papers. The first set of experiments used a cooked starch (Raisaimyl® 40 CF) at loadings from 0.2 to 5 lbs/1000 ft². These starch sheets resulted in no wet bond being formed between the core and the paper after laboratory board formation. Rather, the paper test samples blew off the gypsum core during the drying process, such that the paper was completely removed in the simulated kiln. It appeared that this lack of wet bond was caused by the formation of film surface, preventing water penetration into or through the sheet.

To overcome the film-forming barrier, a non-film forming starch (Staley® WB) was coated onto the sheets at loading from 0.2 to 0.8 lbs/1000 ft². The boards had a mix mode of failure (case IV) meaning that the starch was not able strengthen the weak failure bond zones. The starch was dispersed in solution and drawn down on the paper.

The lab boards made from this paper ranged from 70% (poor) to slightly 30% (good) 2 hour at 90°F / 90% bond, however there was no significant improvement in adhesive bond quality relative to the control boards (40-60% 90°F / 90% bond). The placement of starch at the interface did not show significant improvement in adhesive bond quality. The local variability in the mode of failure may have been caused by uneven distribution of starch. Long [1980] found that adding starch to the gypsum core mix increased the quality of the adhesive bond. Therefore, it appeared that further work is necessary to understand the role of starch for improving the strength of the interface, and thus, the adhesive bond quality. The application of two similar starch products (Raisaimyl® 40 CF and Staley® WB) should not limit the use of other types of starch. Since the exact mechanism of bond has not been determined, the modification made to starch today could easily be used to help form ionic bridges or acts as an aggregate material.

Hydrophobic Surface Coating

Since Pingel [1990] found that limiting the penetration of gypsum core into the board resulted in good adhesive bond, a sizing agent (Cytec® 210) was sprayed upon the surface to form a hydrophobic barrier at the gypsum-core interface. These laboratory boards did achieve a good wet bond, however boards with the sizing barrier performed worse or at the same level as the control boards.

Surface Roughness

The profilometry tests suggested a possible weak connection between roughness and the adhesive bond quality. To explore this affect, two experiments were conducted. First, paper samples were embossed with a wire impression. These wire impressions were made to the paper after the paper had been dried and the structure established. No significant difference in the adhesive bond quality between the control board and gypsum boards made with the wire marked paperboard. However, the laboratory boards produced had a poorer gypsum core strength, which may have led to case III failures.

The second experiment utilized the pilot paper machine at North Carolina State University. The two pilot paper samples were prepared, which varied only in the wet-press application. The pilot trial paper had a mark imparted onto the sheet at the wet press. These samples were found to have acceptable adhesive bond quality.

Both felt-side-away (the intended use of a standard press felt to create a smooth surface) and felt-side-towards (the reverse side of the felt to impart a felt impression) both performed well, reducing the peel of all samples to a value less than 20%. The felt-side-toward the core performed better than felt-side-away because it reduced the adhesive bond quality by approximately 78% compared to the commercial control paperboards. Both paper sample types were successful in reducing the percent of peel. However, the felt marked sheets were able to reduce the peel percentage further. These experiments showed that a relationship exists between the surface characteristics and the adhesive bond.

5.5 HANDSHEET – SET 1

A set of handsheets was developed that changed how moisture was transferred to the paper during manufacturing of gypsum board. The handsheets were made by varying the following papermaking parameters: wet pressing load, ASA size loading per sheet, retention aid loading, wet strength stratification, and sizing stratification of layers. All handsheets had peel percentages of zero, while the control paper had an average rating of approximately 16 percent. A variation of sixteen points is significant. However, even dramatic efforts to change the paper properties did not alter the failure case. Differences between the handsheets and the manufactured paper must be explored to determine why the handsheets had a better adhesive bond. An examination of the difference was conducted to determine the cause of the higher quality adhesive bonds.

Paper made at North Carolina State University performed better than the manufactured paper, as demonstrated in the previous section. Table 5.6.1 shows the comparison of the machine-made paper and the NCSU-made paper physical property results for breaking length, Scott bond, Gurley porosity, and wax pick.

One major difference between the two types of paper was the breaking lengths. The handsheets had a lower breaking length than the machine-made papers. This is important because it was shown in section 5.3 that the MD breaking length influenced the adhesive bond quality. The Scott bond results for the handsheets were in the lower range of the variable than most of the machine made papers. However, the Scott bond values did fall within the range of the machine made papers. The NCSU made papers must have had a lower relative strength (S_p) than the strength of the interface (S_I) and the strength of the core (S_C). In support of an earlier statement (section 5.2), a paperboard made with lower in-plane strength would have a preferred mode of failure (case I). The Scott bond results may indicate a paper with lower z-directional strength would have a better mode of failure (case I).

The Gurley porosity values were mostly lower, but the manufacturing standards only call for a maximum value of porosity; however the values for the NCSU paper samples were generally lower than those for the manufacture paper. The wax pick values varied similarly to the Gurley results. However, the NCSU paper did have a range of results, especially in the case of refined freeness (sample id #s 671, 471, 361, 274, and 183, in which the number labels also correspond to Canadian Standard Freeness levels).

5.6 HANDSHEETS - SET 2 AND SET 3

Based upon the results of the previous section, a second and third set of handsheets was prepared to investigate the effect of wet pressing. This set of tests was designed to vary the densification in the paper. The wet pressing variations would decrease the caliper in the handsheet and result in an increase in apparent density. This would change the ability for air to flow through the sheet and also affect the strength properties. Displayed in Table 5.6.1 are the results of the handsheets trials. Besides the wet pressing loading variation, the basis weight of the sheet was also varied. The difference in basis weight would change the amount of fibrous material for the moisture to transfer through from the gypsum core material.

The results shown in Table 5.6.2 indicate that a slight difference existed between the single-ply handsheets and the multi-ply handsheets for the 2 hours at 90°F/90% r.h. bond quality. Additionally, consistent through both multi-ply and single-ply sheets in the 60 pounds basis weight was that as wet pressing loading increased, the 2 hours at 90°F/90% r.h. bond had an increase in percent peel to the maximum of 100 percent.

Comparing the 60 pound single-ply sheet with the 50 pound single-ply sheet, the bond results for the 50 pound sheet were significantly lower at all levels of wet pressing. The results show that the basis weight played a more significant role in the adhesive quality than the wet pressing load. This experiment agrees with earlier potential correlations with basis weight and apparent density. Further investigation of moisture transfer variables should be investigated to understand which variables play a more important role in the adhesive bond quality.

TABLE 5.6.1 RESULTS OF PHYSICAL PROPERTIES: COMPARISON OF MACHINE MADE PAPERS (A-F) AND NCSU MADE PAPERS COMPARED TO ADHESIVE BOND QUALITY.

Sample	Bond Quality Data 2 Hour @ 20 Hour @			Break Length (km)	Scott Bond		Gurley Porosity (sec)	Wax Pick
	70°F / 50 % R.H. (% peel)	90°F / 90 % R.H (% peel)	90°F / 90 % R.H (% peel)		Anti	MD		
					ft-lb.	ft-lb.		
A	0	36	5	5.37	46.4	47	142	
B	1	52	2	4.75	80.8	83	100	
C	0	79	5	4.99	84.8	97.8	128	10
D	7	86	20	5.77	88.8	91.2	59	
E	11	88	50	5.49	65.2	82.8	123	12
F	7	90	29	5.67	66.6	70.6	106	12
G	9	98	66	5.52	54.2	60	99	12
H	29	99	86	6.2	79.8	80.2	105	18
Pilot		0		4.07	75	82.4		6
671		0		3		56	14	6
471		0		4		73	25	4
361		0		4.39		53.6	35	8
274		0		3.23		48.6	65	18
183		0		4.06		55.6	124	18

**TABLE 5.6.2 RESULTS OF HANDSHEET SET 2 WITH WET PRESS LOADING VARIATIONS
OF SINGLE AND MULTI-PLY HANDSHEETS COMPARED TO THE 2 HOUR AT 90°F/90% ADHESIVE BOND
QUALITY.**

Wet Pressing Loading #/sheet	Multi-Ply 60# /1000 sq. ft (% Peel)	Single 60# /1000 sq. ft (% Peel)	Single 50# /1000 sq. ft (% Peel)
5,000	5	5	10
10,000	5	75	5
15,000	75	75	5
20,000	100	100	15

To understand how important the differences are between the multi-ply and single-ply handsheets an additional experiment was performed. This experiment repeated the wet press loading variation. However, in this set of handsheets the amount of starch used in the core formulation was increased from 7 pounds per thousand square feet to 8 pounds per thousand square feet. In Table 5.6.3, the results show that all handsheets samples yielded zero to five percent peel values. This differs from set two because no increasing trend of percent peel was observed with the increase of wet press loading. This finding reaffirms prior research that the level of starch addition is an overwhelming factor in the quality of the adhesive bond [Pingel, 1990].

With that reaffirmation of previous results, control studies were conducted to ensure the proper level of starch addition. Table 5.6.4 shows the results from control board testing. Again, as the starch level increased, the level of percent peel decreased, thus improving the quality of bond. It is important to note that variability of the data was greater at lower levels of starch. For example, the variability in the 7 pound level was 10 and 35 percent peel and the variability at 8 pound was 60 and 20 percent peel as shown in Table 5.6.4.

TABLE 5.6.3 RESULTS OF HANDSHEET SET 3 WHICH WERE WET PRESS LOADING VARIATIONS OF SINGLE VERSUS MULTI-PLY HANDSHEETS COMPARED TO THE ADHESIVE BOND QUALITY.

Wet Pressing #/sheet	Multi-Ply 60# /1000 sq. ft (%)	Single 60# /1000 sq. ft (%)
5,000	0	0
10,000	5	0
15,000	5	0
20,000	0	0

TABLE 5.6.4 RESULTS OF CORE STARCH LOADING CHANGES OF LAB BOARDS COMPARED TO THE ADHESIVE BOND QUALITY.

Starch (#/1000 sq. ft)	Peel - Side A (%)	Peel - Side B (%)	Average (%)
7	10	35	22.5
8	60	20	40
9	5	0	2.5
10	5	0	2.5

5.7 INVESTIGATION OF SURFACE STRENGTH

Thus far, the relationships between basis weight, apparent density, core starch, and tensile strength have shown some potential for promoting the preferred adhesive bond quality . In addition to these properties, one expects to see a relationship between the internal bonding of the sheet and the adhesive bond quality. In referring to the theory describing four cases of failure, one sees that a sufficiently low adhesive bond strength in paper may be critical in determining case I failures (desired).

Furthermore, the present research has suggested that surface strength may be a potential variable for promoting good adhesive bond quality. As a result of visual observations made during the bond quality test, a study of the surface strength was conducted. Therefore, a set of experiments was carried out, looking at the relationship between wax pick and adhesive bond quality.

The original seven samples of machine made papers (A-H) were tested, and together showed a correlation with the wax pick. Seven additional samples (I-O) were chosen to further investigate the correlation. The wax pick test was chosen because of the nature of the test. It was theorized that the melting wax would approximate the gypsum core slurry, forming a bond with the paper. Higher values for wax pick indicate a stronger paper, while lower values for wax pick indicate a weaker surface strength.

Table 5.7.1 shows the results of wax pick versus the bond quality data at the three conditions. Peel results displayed by an F (red) were the samples produced on a Fourdrinier type paper machine. Peel results displayed C1 (green) were the samples produced on the same cylinder board machine as discussed earlier. The two remaining C2 (blue) samples were created on a separate cylinder board machine with different production variables than the green cylinder board machine. A general trend can be observed that Fourdrinier papers produced more desirable bond quality peel percentage than the cylinder board papers. Also, the wax pick of Fourdrinier papers was observed to be lower, meaning a lower surface strength to the paper. This is important because at lower surface strength the paper appears to be weaker than the adhesive bond strength, leaving the paper to tear apart somewhere within the paper sheet (case I).

It is also important to discuss the correlation of the wax pick results to the various bond quality conditions. The 70°F/50% r.h. (green diamonds), 2 hours at 90°F/90% r.h. (red, boxes), 20 hours at 90°F/90% r.h. (blue triangles) bond results are plotted versus wax pick in Figure 5.7.1. The 70°F/50% r.h. condition occupies the lower area of the graph, as shown by the lower oval. The 2 hours at 90°F/90% r.h. bond occupies the upper area of the graph shown by the upper oval. For the 70°F/50% r.h. bond, the area of indiscriminant values occurs at higher wax pick values, while the 2 hours at 90°F/90% r.h. humidified bond has its indiscriminant values at lower values of wax pick.

TABLE 5.7.1 A TABLE SHOWING THE WAX PICK RESULTS COMPARED TO THE THREE PAPER MACHINE SETUPS.

Sample	Machine Type	Bond Quality Data			Wax Pick
		70°F / 50	2 Hour @	20 Hour @	
		% R.H. (% peel)	90°F / 90 % R.H (% peel)	90°F / 90 % R.H (% peel)	
I	F	1	14	2	9.80
A	C2	0	36	34	11.80
B	F	1	53	2	10.25
J	F	1	56	2	9.40
C	F	0	79	5	10.33
L	C1	1	85	16	9.20
E	C1	7	90	29	12.40
N	C1	6	90	37	14.00
O	C1	80	93	93	14.40
F	C1	7	96	20	13.00
G	C1	9	98	66	12.80
D	C2	11	99	63	12.40
H	C1	29	99	86	16.00
P	C1	60	99	85	16.00

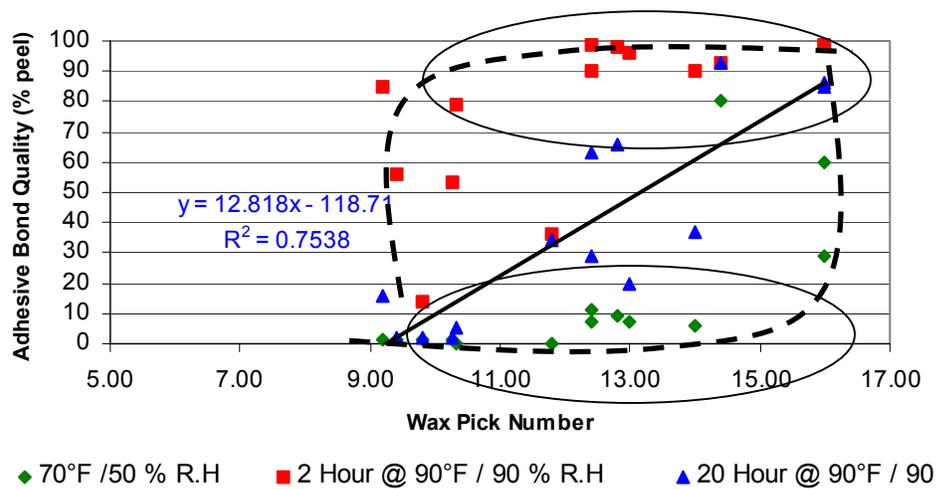


FIGURE 5.7.1 RESULTS OF WAX PICK TESTING FROM THREE THE THREE TEST CONIDITIONS COMPARED TO THE ADHESIVE BOND QUALITY.

It is important to illustrate the following dynamics believed to be occurring with the 20 hours at 90°F/90% r.h. humidified bond . Three states are believed to be occurring: lower surface strength, medium surface strength, and high surface strength values. At the lower surface strength values, 9 – 11 wax pick, the adhesive bond was believed to be stronger than the paper bond. Therefore, the paper will tear apart leaving paper coverage on the core surface (case I). At higher strength values, 15-17 wax pick, the paper strength was believed to be stronger than the adhesive strength, resulting in paper being completely removed from the gypsum core (case II). At medium values of surface strength, 12- 14, the adhesive bond strength and the paper strength were in a varying state of nearly equal strength where at some point the adhesive strength was lightly greater and at other the paper strength was slightly greater causing variation in the percent of core revealed (case IV).

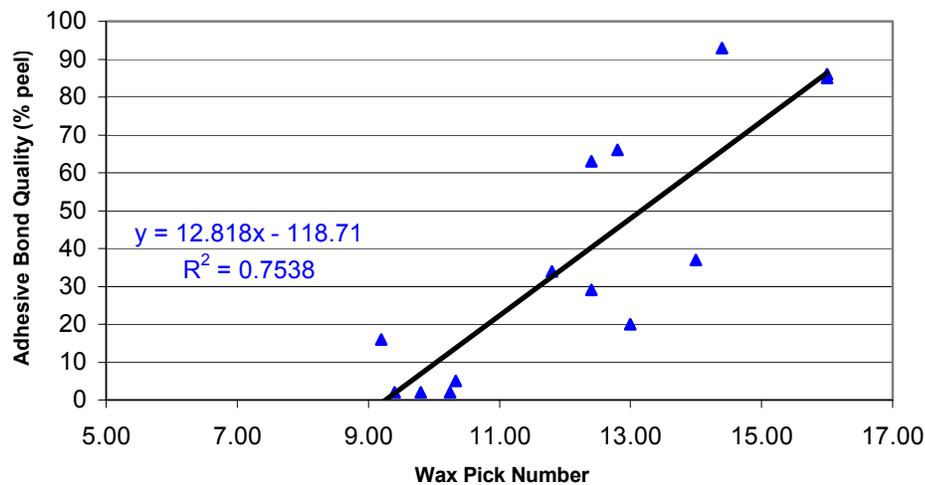


FIGURE 5.7.2 EXTRAPOLATED ZERO-POINT WAX PICK FOR USE IN COMMERCIAL QUALITY CONTROL.

Since a correlation exists between the wax pick and the adhesive bond quality, the test may be used as an industrial quality test. The ability to determine if the paper will have a desired mode of failure before conversion of paper could help both gypsum board plants and paper plants. Control limits would need to be established for wax pick values. Based on this research, a value (zero-point wax pick) could be found to achieve a zero percent peel and a good adhesive bond quality (case I). The zero-point wax pick value was extrapolated and is approximately 9.26, as shown in Figure 5.7.2 where the trend line intersects with the x-axis.

It is hypothesized that there is uneven moisture content across the paper, causing two-hour humidified bond values with low picks to have poor adhesive bond quality. During moisture absorption, or transmission in this case, the properties of the paper will be in a state of constant changing until the equilibrium moisture was reached. The first experiment was designed to measure the degree of surface strength change as a result of humidification.

TABLE 5.7.2 RESULTS OF WAX PICK CHANGE DUE TO 2 HOURS AT 90°F/90% R.H. HUMIDIFICATION.

Sample	70°F/50% R.H. Wax Pick	2 hours at 90°F/90% R.H. Wax Pick	70°F/50% R.H. to 2 hours at 90°F/90% R.H. Difference
G	12.8	8.5	4.5
H	16.0	11.5	4.5

This experiment measured the amount of change occurring between the 70°F/50% r.h. condition and the 2 hours at 90°F/90% r.h. condition. Of the remaining samples available, two samples with different 70°F/50% r.h. wax picks were chosen for 2 hours at 90°F/90% r.h. The results show in Table 5.7.2 that the wax pick values for both samples were lowered by 4.5 wax pick numbers from the original 70°F/50% r.h. condition. Based on the zero point wax pick, of 9.26, sample G should have a good adhesive bond and sample H should have a poor adhesive bond quality, if the 70°F/50% r.h is a measure of absolute strength or only applicable at the 70°F/50% r.h condition. Referring back to Table 5.7.1, both samples G and H performed poorly at the 2 hours at 90°F/90% r.h. condition.

Of the remaining samples available, three samples with different 70°F/50% r.h. wax picks were chosen for 20 hours at 90°F/90% r.h.. The results show, in Table 5.7.3, that the wax pick values for both samples were lowered in the range of 5.5 to 7.7 wax pick from the original 70°F/50% r.h. results.

TABLE 5.7.3 RESULTS OF WAX PICK CHANGE DUE TO 20 HOURS AT 90°F/90% R.H. HUMIDIFICATION.

Sample	70°F/50% R.H. Wax Pick	20 Hour Wax Pick	70°F/50% R.H. to 20 hours at 90°F/90% R.H. Difference
I	9.8	4.3	5.5
F	13.0	6.5	6.3
H	16.0	9.7	7.7

Based on the zero-point wax pick, of 9.26, sample I and F should have a good adhesive bond (case I) and sample H should have a poor adhesive bond quality (case IV), if the 70°F/50% r.h is a measure of absolute strength or only applicable at the 70°F/50% r.h condition. Referring back to Table 5.7.1, both samples H and I performed well at the 20 hours at 90°F/90% r.h. condition and sample H performed poorly. Although the 2-hour bond test did not show the zero wax pick value to be true, the 20-hour sample does.

Given the results of the zero point wax pick, one must consider that a material with a lower surface strength or low z-directional strength, as indicated by wax pick, gives a better adhesive bond quality results (case I). This result, as discussed in section 4.1, proves that the desired plane failure occurs in the sheet of paper as opposed to the paper core interface and this is not a measure of strength of the gypsum bond. However, is the surface strength only important or is z-directional strength important as well?

5.8 DEBONDER APPLICATIONS ON MACHINE-MADE PAPER

Because surface strength and, potentially, z-directional strength are important, the use of a debonding agent was proposed to manipulate the z-directional strength of the paper. The application of a debonding agent on the surface of the paper when remoistened should disrupt the fiber-bonding network of the paper, changing the overall strength of the paper during the gypsum manufacturing process. The debonding material was applied to the paper surfaces at four levels.

Table 5.8.1 displays the preliminary test results, showing that a positive improvement in adhesive bond quality was observed when the coating level increased to above 1 percent. It is important to note that the major improvement occurred somewhere between the 0.1 percent and the 1 percent coating level. The peel percent difference between the 1, 5, and 10 percent level was not considered to be significant. Two possible mechanisms may be occurring as a result of the debonding.

TABLE 5.8.1 RESULTS OF USING A DEBONDING AGENT #692 ON THE SURFACE OF PAPERBOARD TO EFFECT THE ADHESIVE BOND STRENGTH.

Coating Level (%)	Peel (%)
0.1	27.5
1	2.5
5	2.5
10	0

First, the debonding agent may have allowed better penetration of the gypsum slurry into the paper, resulting in an improved wet bond or better potential for gypsum crystal formation into the paper. Second, it was observed that the debonder caused a wet bond crumbling affect within the paper structure when performing the wet bond measure. This could have allowed for better permeability of moisture during the initial phases of drying by changing the nature of the pore structure to allow better penetration of the moisture.

Given the results of wax pick and the debonding agent, further study is necessary to determine if a paper with low z-direction strength is good for gypsum wall board. However, this paper must also maintain high tensile strength properties [Johnstone, 1984; Balzer and Mohen, 2000]. This research has shown the potential for creating a paper with low z-direction strength and a paper with high in-plane modulus. It may also be possible to lower the strength of the paper and still meet industry standards.

5.9 MOISTURE ANALYSIS

With the results of wax pick, Figure 5.7.1, a moisture time relationship is occurring during the adhesive bond quality test. To understand this relationship, a set of experiments was designed. Samples were taken from a standard condition of 70°F/50% r.h and placed in a humidity chamber of 90°F/90% r.h.. Figure 5.9.1 shows the absorption curve for the base paperboard with time. The data show that providing a barrier to moisture absorption on one side of a paper sheet (i.e. contact paper) has little influence on the ratio of moisture absorption time. It is also important to note that the samples are picking up moisture until approximately 1000 minutes (16.7 hours). As seen in Figure 5.9.2, the rates of moisture pickup, shown as a ratio of final equilibrium moisture content are virtually identical.

Figure 5.9.3 shows the relationship of the starch-coated paperboard with time. The data show that all three samples proceeded similarly to the equilibrium moisture condition. It is also important to note that the samples are picking up moisture until approximately 1000 minutes (16.7 hours). This supports the theory hypothesized early that the 2-hour test condition has not reached an equilibrium condition and it is conceivable that the moisture content within the sheet is not even and could cause the variability in adhesive bond quality and where the plane of failure if occurring within the gypsum board. Additionally as seen in Figure 5.9.4, the rates of moisture pickup, shown as a percent of final equilibrium moisture content are virtually identical.

When comparing the base paperboard with the starch-coated paperboard (Table 5.9.5), the rates of moisture pickup for the samples are all similar. This information shows that when exposed to moisture, the starch coating on the paper gains moisture at the same rate and that moisture pickup is independent of the paper media. Given the model presented, this means that at least two of the three interfaces can be treated as independent bodies with respect to moisture.

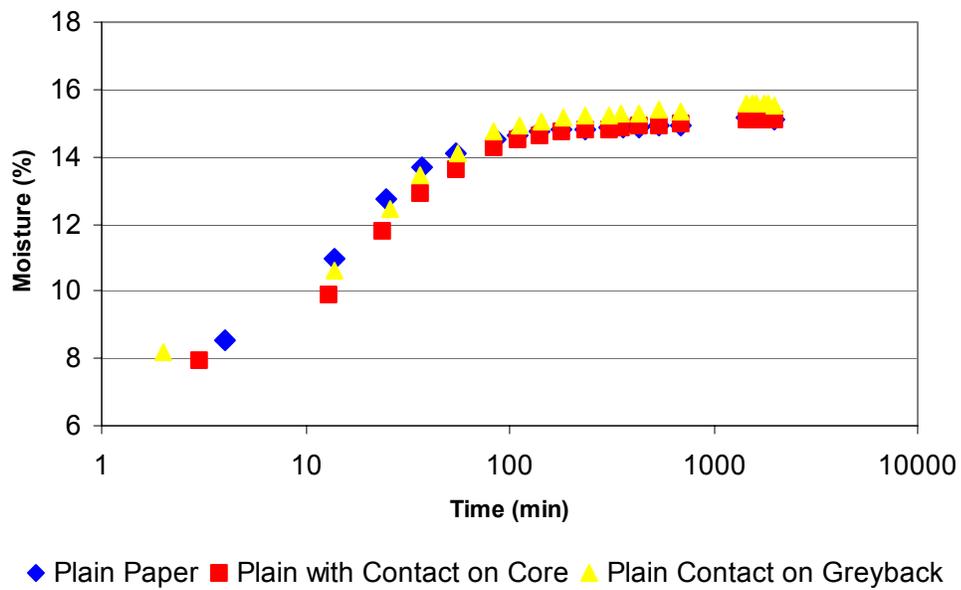


FIGURE 5.9.1 MOISTURE PICK UP RATE RESULTS FOR PLAIN PAPERBOARD AND PAPERBOARD WITH CONTACT PAPER BARRIER.

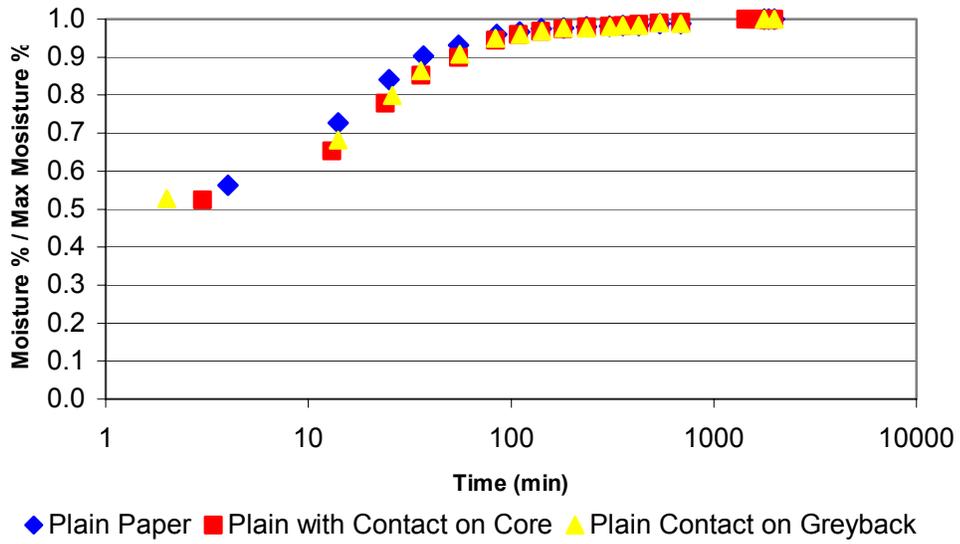


FIGURE 5.9.2 RATIO OF MOISTURE OVER THE MAXIMUM MOISTURE FOR PLAIN PAPERBOARD AND PAPERBOARD WITH CONTACT PAPER BARRIER.

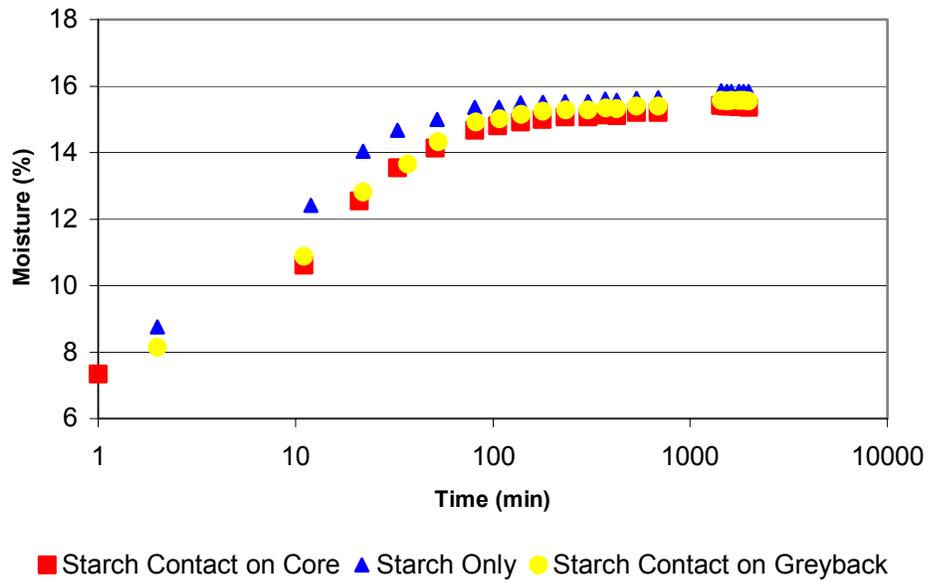


FIGURE 5.9.3 MOISTURE PICK UP RATE RESULTS FOR STARCH COATED PAPERBOARD AND PAPERBOARD WITH CONTACT PAPER BARRIER.

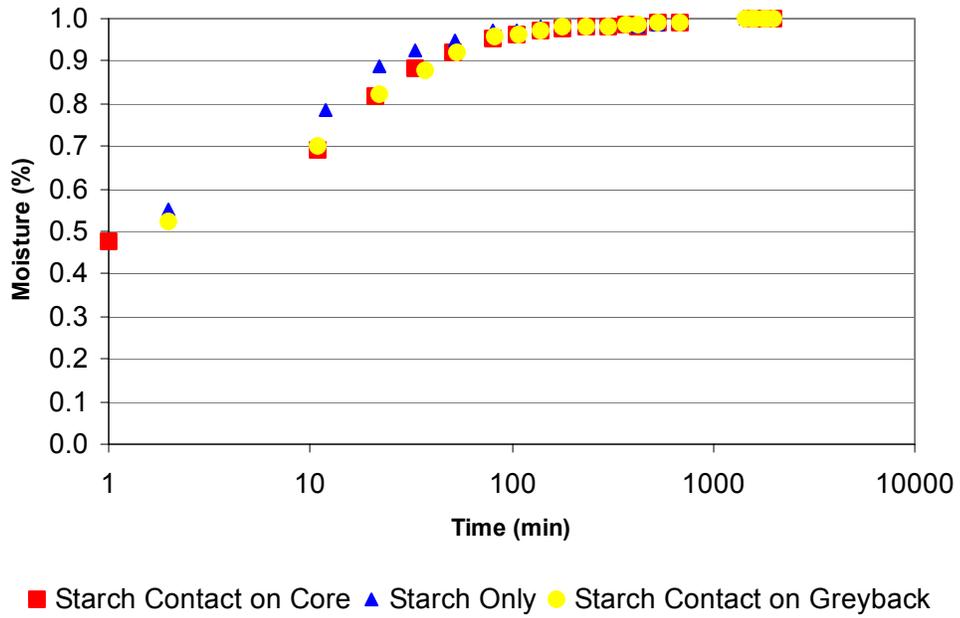


FIGURE 5.9.4 MOISTURE PICK UP RATE (SHOWN AS PERCENT OF MAXIMUM MOISTURE) RESULTS FOR STARCH COATED PAPERBOARD AND PAPERBORD WITH CONTACT PAPER BARRIER.

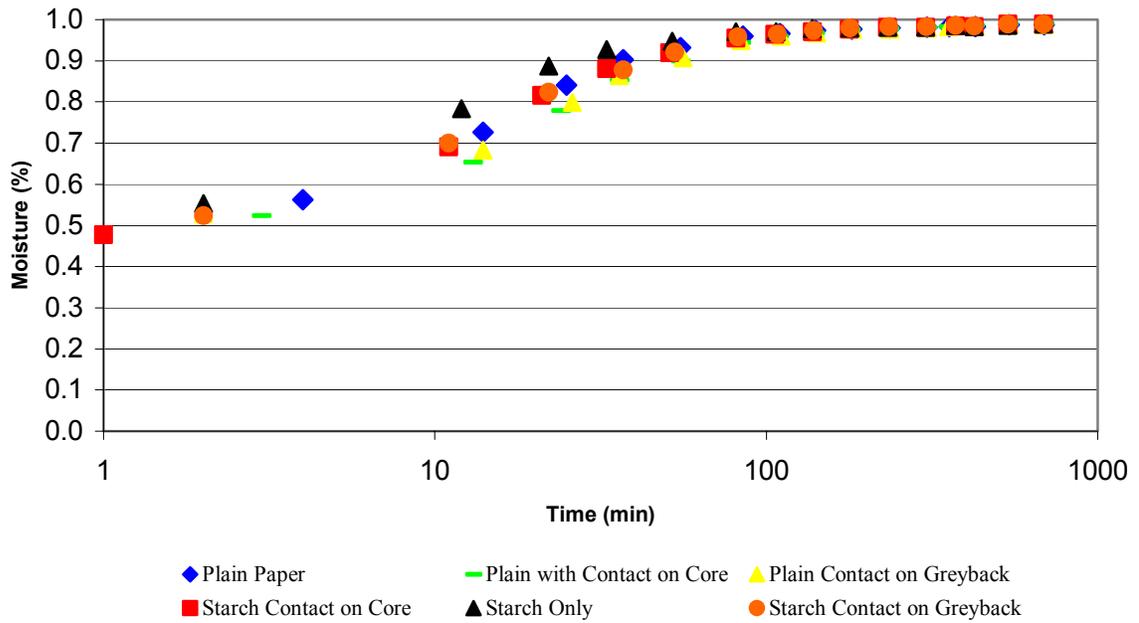


FIGURE 5.9.5 MOISTURE PICK UP RATE COMPAIRSON FOR ALL SAMPLES.

5.10 SUMMARY

The results of the moisture study indicate that a dynamic moisture condition is occurring within the sheet of paper during the 2-hour humidified bond condition. This dynamic condition ends when the paper achieves its equilibrium moisture condition after approximately 1000 minutes (16.67 hours) or during the 20-hour humidified bond test. This information supports the earlier work that the 70°F/50% adhesive bond quality test and the 20-hour humidified bond test moderately correlate to each other. Furthermore from the moisture experiments, the data shows that the various zones (as proposed in the model of adhesion) will absorb moisture at independent rates.

The concept of manufacturing a high in-plane strength sheet with low z-directional strength is moderately supported. The necessity to produce a high in-plane strength sheet for gypsum board has been shown (section 2.4). However, the data from this research, such as basis weight, low density, and low breaking length (section 5.3 & 5.5), makes it difficult to support a high in-plane sheet.

However, a high in-plane sheet with a low z-direction strength may be able to meet the gypsum wallboard standards and achieve a high quality adhesive bond. Based on the wax pick data, the z-directional strength of the paper immediately adjacent to the surface plays a major role in the failure or the adhesive bond quality (section 5.7). The wax pick relationship was further supported by the application of a debonding agent to disruption the paper at the adjacent surface (section 5.8). Further work is necessary to show that an overall lower z-directional strength throughout the paper would be equally effective in achieve a good adhesive bond. However, this work shows that a lower strength paper immediately adjacent to the surface can have the preferred plane of failure and a good adhesive bond quality.

Given that a lower strength paper immediately adjacent to the surface can have the preferred plane of failure and a good adhesive bond quality, the ability to determine if the paper will have a desired mode of failure before conversion of paper could help both gypsum board plants and paper plants. This work has shown that the wax pick can be used as an industrial control test. The control limits would need to be further established however, this research has shown that a value (zero-point wax pick) could be found to achieve a zero percent peel and a good adhesive bond quality (case I). The zero-point wax pick value was approximately 9.26, as shown in Figure 5.7.2.

6 Conclusions

This research has shown that the proposed modes of adhesive bond failure are credible. This work has shown that the twenty-hour bond values were the most reliable measure of adhesive bond quality followed by the 70°F/50% r.h. The two humidified bond condition shows that a different type of failure is occurring in the dynamic condition. During moisture sorption, a dynamic nature until equilibrium moisture content has been reached.

The moisture sorption of the three adhesive bond interfaces has been shown to be independent. The rate of moisture pickup for paperboard, starch, and starch-coated paperboard are similar. Therefore, it has been shown that the three adhesive zone strengths can behave differently.

Wax pick investigations led to a good correlation between surface strength and adhesive bond failure. A zero point wax pick was extrapolated to a value of 9.26. Paperboard made with strength less than 9.26 or as a result of humidification loses strength to a value less than 9.26 resulted in a favorable adhesive bond quality. Alternatively, application of a debonding agent to the surface of the paperboard was able to sufficiently weaken the paper to cause a favorable plane of failure and a good adhesive bond quality.

The investigation of NCSU handsheet performance and machine-made paper performance showed that a difference between the handsheet paper and the machine made paper existed. An understanding of the mechanism that results in the handsheet and machine made paper difference was not determined.

Multi-ply handsheets were an important aspect of gypsum board research, since most commercial manufacturers currently have cylinder board paper machines. The multi-ply sheets performed as well as the single-ply sheet in the performed research, meaning ply bond failure was not the direct cause of the higher quality of adhesive bonds during this research.

This research confirmed prior research as to the importance of starch within the core formulations. The absence of starch in the core mixes led to poor formation of adhesive quality within the laboratory boards. Also, all conditions with a large amount of starch added to the core formulations created a high quality adhesive bond as measured by current described techniques. However starch coated paper, uncooked and cooked, were shown to decrease adhesive bond quality.

Several other papers properties have been shown to affect the adhesive bond quality of gypsum board. The effects of wet pressing paper appeared to be directly linked to the ability to form a good adhesive bond between the paper and the gypsum core. Increased MD breaking length was shown to negatively affect the adhesive bond quality. A lower density sheet was shown to have an improved bond performance. Finally, paper at lower basis weights performed better.

7 Recommendations

Critical to the success of future research is the accurate measurement of the force required to break in the adhesive bond between paper and the gypsum core. Therefore, a primary recommendation is to modify current peel force testers for the evaluation of gypsum board. It is believed that the current method of evaluation does not fully describe the quality of the adhesive bond; however a visual inspection or plane of failure evaluation should not be dismissed. Only in conjunction with the peel force tester and a visual inspection can the most accurate understanding of the adhesive bond be observed.

Water is a key component in the formation and maintenance of good adhesive quality. Therefore, it is recommended that a moisture study to further evaluate the dynamic nature of the penetration of water into the paper gypsum surface, similar to the conditions that the gypsum board may encounter during end use. Because moisture transfer into the paper creates a dynamic affect until equilibrium is reached, it will be important to develop a new set of criteria for future testing based upon the moisture work. The current method of measuring 2-hour humidified bond may be at a critical junction to the dynamic change in paper properties during humidification. Therefore, it is important to evaluate the adhesive bond quality at various stages of moisture pickup from the dynamic to the equilibrium state and compare those results to the standard condition room.

A relationship between the surface strength has been shown to be a good predictor of adhesive quality. The nature of this relationship suggests that wax acts similarly to the gypsum core material in creating a bond to the paper. It is essential to further test this relationship with the potential that a quality control procedure can be instituted during manufacturing. The application of debonding agents and their mechanism of improvement needs to be investigated. Based on surface strength and debonding results, it appears that a paper with high in-plane modulus and low z-directional strength may be appropriate design parameters for gypsum paperboard.

A relationship between the internal bond strength and the adhesive quality has been loosely established. It is recommended that further characterization of the internal bond be explored. Koubas and Karan [1995] have studied three methods (z-direction tensile strength, Scott Bond, and delamination test) in evaluating the internal bond of paper and board. Core starch played a vital role in the development of good adhesive bond. Starch usage during testing must be considered a key variable that can overwhelm other potential variables. Care should be used to establish appropriate starch usage. Based on moisture work and core formulations, further work is necessary to understand the relationship of starch within the adhesive bond interface.

The original handsheet study provided no information about the ability for sizing, ASA stratification, wet strength, and refining to affect adhesive bond quality. These properties should be retested with the new standard methods for laboratory production.

Additionally, the manipulation of the physical surface by wire impression was not successful because failure of the bond wasn't achieved under original test procedure. A reevaluation of physical surface properties, by wire impression or another method, is necessary to evaluate the potential shown from the R_a correlation in the Profilometry.

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