Recycling Evaluation of Newly Developed Environmentally Benign Pressure Sensitive Adhesive for Postage Applications

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Abstract

The main objective of the United States Postal Service's (USPS) program for environmentally benign pressure sensitive adhesive (PSA) is to develop postage stamp adhesives that do not adversely affect the environment. The goal is to develop PSA stamp products that can be successfully recycled into fine paper products in a typical recycling facility. This paper describes the development of the final specification and approved product list for PSA stamp production. Environmentally benign PSAs are a form of adhesive that places no significant additional burden on plants that are using recycled fiber. As a result of an initiative by the USPS, a team has been working cooperatively to help solve the problem of PSAs not being recyclable in recovered paper. The team consists of the USPS; the USDA Forest Service, Forest Products Laboratory (FPL); Springborn Testing and Research (STR); and industry representatives. Industry members include papermakers, paper recyclers, paper collectors, equipment manufacturers, and adhesive and chemical suppliers. The team's goal is to work with industry to develop environmentally benign PSAs for postal applications that will fulfill both USPS specifications and be compatible with the USPS environmental strategic plan and will place no additional burdens on plants that use recycled fiber. In 1995, the USPS sponsored a conference to which the adhesive industry was invited to participate in the USPS Environmentally Benign Pressure Sensitive Adhesives for Postage Stamp Applications Program. Several companies participated by submitting adhesive samples. To determine whether an adhesive was recyclable, a protocol needed to be developed. The

result of this work led to a three-tier evaluation process with the development of laboratory, pilot-scale, and mill-scale recycling protocols. The laboratory and pilot-scale protocols were developed and implemented at STR and FPL, respectively. Both the laboratory and pilot-scale protocols will be incorporated into a new USPS recyclable PSA stamp specification. All adhesive samples participating in the program that successfully meet all the qualification requirements of the specification, including the newly developed recycling protocols, will be placed on the USPS qualified PSA stamp products list. The developments by FPL and STR of the laboratory and the pilot-scale protocols are briefly described in this paper. More detailed reports are described in the references at the end of this paper.

Introduction

Since 1990, the self-adhesive stamp volume has increased continuously each year. In 1997, 82% of the stamps in the USPS program were produced in self-adhesive formats. The USPS has had to respond to many customers' concerns regarding the environmental impact of self-adhesive postage stamps. The pressure has been mounting to find an environmentally benign PSA for use in the USPS stamp program. In response, the USPS prepared a concept paper identifying their concerns and goals, conducted an industry survey, and developed a statement of work. In 1995, the USPS awarded a contract to STR entitled "Research & Development of an Environmentally Benign Pressure Sensitive Adhesive for Use on Postage Stamps".

The main objective of the USPS's environmentally benign PSA program is to develop postage stamp adhesives that do not adversely affect the environment. The current research and development (R&D) effort is concerned with PSA material in both the preconsumer and the postconsumer waste stream. The preconsumer waste materials originate from laminate converting, stamp printing and finishing, and disposal of nonspecification material. The postconsumer waste material consists primarily of stamped envelopes found in office paper waste streams.

The USPS goal is to develop PSA stamp products that can be successfully recycled into paper products in a typical recycling facility, particularly those plants supplying pulp for printing and writing (fine) grades of paper. The newly developed stamp and stamp materials should not contribute a significant additional burden on paper recycling mills equipped to handle high quality. de-inking grades of waste paper.

Major Issues

The main obstacle in this effort is that typical PSA materials, by nature, are tacky. When introduced into the paper recycling stream, the adhesives are broken down into small particles called stickies. These stickies cause problems in the paper recycling process. They adhere to wires. screens, and felts and cause specks or tears in the final paper product. Downtime for cleaning of recycling equipment is time consuming and costly, so paper manufacturers are reluctant to accept wastepaper that has a high PSA content.

Another problem is that the industry does not have adequate information or test data to justify conducting a mill-scale trial. Our effort invites the industry to jointly participate in the development, evaluation, and laboratory- and pilot-scale testing of PSA samples submitted. The USPS wants to ensure that a systematic approach is used to create and analyze both preconsumer and postconsumer waste data, particularly with regard to the stickies problem that has concerned the industry for many years.

In addition to being recyclable, the adhesive used in PSA stamp products must first meet stringent performance requirements of the USPS. These requirements include permanence to envelop substrates and ability to withstand long-term aging (long shelf life). To best solve the problems of conflicting performance requirements, the research began by investigating all factors that influence the final production of the stamp, as well as mail processing and recyclability. At the 1995 USPS-sponsored conference, participants from the adhesive industry were solicited to work cooperatively with the USPS to develop the desired adhesive. The response was very positive, with more than 16 companies offering to participate. In July 1997, a second conference was conducted to report the progress of those companies.

Team Effort

The USPS wanted to encourage a focused, industry-wide approach for meeting the requirement for an environmentally benign PSA stamp. They started promoting the vertical team concept in 1996. With USPS encouragement, various industry participants formed vertical teams. Team members represent adhesive suppliers, stamp printers, converters, face and liner paper suppliers, recycling mills, testing laboratories, and equipment manufacturers. The teams are working on novel solutions to the problem of a recyclable PSA product that meets the performance requirements of the USPS. As many as 27 adhesives have been submitted for independent evaluation of their performance and recycling properties.

Protocol Development

A three-tier recycling protocol was developed that includes evaluating candidate stamps and stamp materials for recyclability at the laboratory and pilot scale and, in the future, at the mill level. Criteria for acceptance of these protocols were agreed upon with the cooperation and input of several paper recycling mills. The results that are presented in this paper, based on these test methods, are clear indications of the recyclability of these candidate adhesives. An effort to thoroughly test each of the adhesive samples is on-going. Research findings and team approaches will be presented during the TAPPI Recycling Conference in March of 1998 in New Orleans, LA. Acceptable materials will then have to undergo extensive converting and printing trials, as well as a mill-scale test procedure.

Final Product

At the conclusion of this project, a specification, including recycling requirements, will be prepared. To qualify as USPS stamp construction material, all future stamp products will have to conform to this new specification. In addition to the postage stamp, the USPS uses PSA tapes and labels for a variety of applications. It is estimated that the USPS uses more than 250×10^9 in^2 (16 1.29×10^9 m^2) of PSA stamps and labels per year, which is approximately 14% of the total U.S. label market. The USPS also expects to apply the same recyclable adhesive technology developed for stamps to other postal label products in the future. A discussion of both the laboratory-scale and the pilot-scale protocols follows.

Laboratory-Scale Protocol Development

Since 1994, STR has been working with the USPS to develop PSAs for postage stamp applications that will not adversely affect the environment. To successfully achieve this objective, a complete understanding is required of two areas: (i) postage stamp requirements including performance. production, mail processing, and philatelic and (ii) paper recycling issues as they relate to adhesive contaminates, furnish, and end products.

To achieve the in-depth knowledge required in these areas, paper recycling and adhesive literature was reviewed, experts from the paper and adhesive industry were interviewed, equipment manufacturers were visited, and information from the USPS was studied. Among the numerous information, two important findings were uncovered:

- Several adhesive manufacturers were actively working on developing recyclable adhesives, so the technology was available and in some cases commercially available.
- No universally accepted recycling test method or protocol was available; all recycling procedures reviewed seemed to be dependent upon the recycling mill, the end products being made from the recycled pulp, or the adhesive company designing the adhesive.

To take advantage of the recyclable adhesive developments in progress, the USPS invited the adhesive industry to participate in the USPS Environmentally Benign Pressure Sensitive Adhesives for Postage Stamp Applications Program at the USPS-sponsored conference in 1995. The adhesive industry was asked to submit samples of adhesive products that would meet the current USPS performance specifications as well as be recyclable. The response to the invitation was encouraging, with many companies ultimately participating in the program.

It was found that there were differences in the ways the adhesive companies participating in the program were defining and testing adhesive recyclability. Furthermore, there was little communication between the adhesive industry developing the product and the recycling industry. Fostering the cooperative efforts of the adhesive and paper recycling industries as well as other industries has become a key strategy in developing a consistent protocol for evaluating the recyclability of PSAs for postal applications and achieving the USPS objective of developing a recyclable adhesive.

In developing a laboratory recycling protocol, all resources, including the literature and cooperative efforts of the adhesive industry participants and the recycling industry, were utilized. The USPS wanted to qualify the entire stamp laminate construction (face paper, adhesive, and release liner) as recyclable; therefore, the recycling protocol incorporated both preconsumer (with liner) and postconsumer (stamp, no liner) stamp waste. The result of this development work has led to a three-tier evaluation process with the development of laboratory, pilot-scale, and mill-scale recycling protocols. The laboratory and pilot-scale protocols are being developed and implemented at STR and FPL, respectively, for the USPS. Both the laboratory and pilot-scale protocols will be incorporated into a new USPS recyclable PSA stamp specification. All adhesive samples participating in the program that successfully meet all the qualification requirements of the specification, including the newly developed recycling protocols, will be placed on the USPS qualified PSA stamp products list.

This part discusses the development of the laboratory protocol and provides insight to the design and selection of parameters and methods used that define the current protocols. Also included are results of preliminary efforts to better understand the limits of the laboratory protocol and to better understand the influence of changes in various process conditions on the final results of unprinted laminate samples.

Discussion

Several challenges were encountered in the development of the laboratory recycling protocol for the Environmentally Benign PSA Stamp Project. The first was to identify the typical processes and conditions on which to base the protocol so it would simulate actual recycling mill practices. Based upon the process selected, we needed to define the parameters to be measured and the acceptance criteria for being considered a recyclable adhesive product for postage stamp applications. The next step was to implement, evaluate, and refine the protocols.

Another challenge was to bring together experts from various fields, especially the adhesive and recycling industries, to exchange information on stamps, adhesives, and paper recycling. By promoting the information exchange, a better understanding of the entire stamp lifecycle was achieved, adhesive manufacturers are better able to design a recyclable adhesive, and the recycling protocol will better represent the current mill practices.

The following sections discuss the development stages leading to the USPS laboratory recycling protocol used to evaluate candidate stamp materials as part of the testing phase of the USPS environmentally benign PSA stamp program.

Adhesive Evaluation

The testing phase of the USPS environmentally benign PSA stamp program involved evaluating stamp samples submitted by the adhesive companies participating in this program. The samples consisted of unprinted laminate constructions composed of face paper (containing a water-soluble tie layer), PSA, and release liner. The adhesives used in the samples included three main classes: acrylic or modified acrylic, rubber, and polyester. The overall sample evaluation was based upon two criteria: adhesive performance and recyclability. The stamp adhesive must perform satisfactorily as a stamp first. Therefore, testing began with the PSA stamp performance properties based upon a modified version of the USPS-P- 1238B PSA stamp specification. The three properties that needed to be met include permanence of the adhesive bond between a stamp and envelope substrate, aging of stamp materials in three environments (-40°C (-40°F), 70°C (158°F), and 38°C (100.4°F) with 90% relative humidity (RH)) and evaluating each material for undesirable visible or physical changes after these environments, and the ability of the stamp affixed to an envelope to be removed by soaking in water. A previous paper details the specific performance testing, ranking, and final results for two rounds of samples submitted (1).

In summary, 27 adhesive samples from 13 participating companies were evaluated in the latest round of PSA performance testing. Based upon the results of this testing, 14 of the 27 samples were selected for recycling evaluation.

Laboratory Recycling Protocol Development

Protocol Model-Printing and Writing Grade: Since a recycling test protocol was not established at the onset of the testing phase, a protocol needed to be defined, documented, and evaluated before the testing phase of this program could be completed. The main goal for the design of the recycling protocol was to provide a controlled method for evaluating the effects of PSAs used in the construction of the postage stamps on the paper recycling process and end products.

In designing a recycling protocol for PSA postage stamps, consideration was given to the wastepaper infrastructure. The possible waste streams in which stamps may ultimately be found and the possible end products into which the wastepaper may be made were of primary concern. The goal was to be able to make high-quality, value-added products from the wastepaper containing stamps. For this reason, the most logical waste stream on which to model the USPS recycling protocol was the recycling of mixed office waste (MOW), containing stamped envelopes, into high-grade printing and writing papers. Therefore, the development of the protocol was based upon the recycling techniques used in the fine-grade deinking recycling mills. In this waste stream, the fiber contains a high. chemical pulp content.

Recycling Industry Participation: Among the proprietary processes and variability of the recycling practices at mills recycling MOW into fine-grade paper or tissue, certain unit operations and processes were found to be consistent. These operations included pulping, coarse and fine screening, forward and reverse cleaning, flotation, and washing.

Furthermore, from the discussions with the paper recycling industry (printing- and writing-grade products), two primary philosophies in adhesive contaminate removal during recycling were identified. The first was the size reduction of the contaminate particles using somewhat severe pulping conditions. The theory was that particles could be made small enough to be removed by flotation and further removed from the wastewater using clarification. Adhesives designed for this removal technique were primarily dispersible types and emphasis was placed on the size of dispersed adhesive particles as a result of pulping. Negative factors associated with dispersible adhesives included the possibility of reagglomeration later in the paper recycling process and build-up effects (especially in closed-loop systems). In addition, the dispersible adhesive types were not successful at meeting the USPS high humidity aging performance test.

The second method was the use of low pulping temperatures and mild agitation to minimize breaking up of the adhesive contaminates into small particles. In this case, removal of the adhesive particles is performed with pressure screening. The ideal adhesives for this method of removal are those designed to remain fairly large during pulping and relatively nontacky while wet. The recycling industry members interviewed favored adhesives that could be screened from the system early in the recycling process as the primary means of contaminate removal.

The USPS laboratory recycling protocol was designed to evaluate the recyclability of adhesives in the feed-through screening, leaving the pilot-scale protocol to address other separation steps including density separation, flotation, and washing. However, currently, developments are underway to incorporate flotation studies into the laboratory protocol for predicting removal of small and finely dispersed adhesive and ink particles to evaluate printed stamp materials in the future.

The goal was to develop laboratory and pilot-scale recycling protocols that are compatible with one another and meaningful to the recycling mills; therefore, the USPS, STR, and FPL jointly invited representatives from several printing-, writing-, and tissue-grade recycling mills to participate in the development and optimization of both the laboratory and pilot-scale recycling protocols. Several meetings and demonstrations of both recycling protocols were, and will continue to be, held with the industry representatives present.

The current revision of the laboratory protocol (LRP-3) is presented in Appendix A. Since the intention of the USPS is to be able to recycle the entire stamp product, both preconsumer (including release liner) and postconsumer (no liner, for example, stamped envelopes) stamp materials were included in the protocol. To study the effects of the adhesives on recycling, the contaminants, or stickies, in this protocol are solely from the stamp adhesive; no other adhesives, tacky materials, or printed materials are present in the furnish. The postconsumer feed stock consisted of a blend of 95% copy paper and 5% unprinted stamp stock and is denoted as Configuration A in the protocol. The preconsumer feed stock includes 10% stamp laminate (with release liner) and 90% copy paper (Configuration C). For the standard PSA stamp booklet construction with a 130 g/m^2 (80 lb) release liner, the pre- and postconsumer furnishes contain approximately equivalent levels of adhesive.

The high level of stamp stock (5%)in the furnish was selected to ensure a margin of safety when analyzing the screened adhesive product. A blend of 5% stamp stock, with an estimated adhesive coat weight of 24 g/m^2 (0.0045 lb/ft^2), and 95% copy paper contains approximately 1% adhesive by weight. a level much greater than would be typically expected in the furnish used at a mill recycling MOW. In comparison, the adhesive on 645.16 mm^2 (1 in^2) of this same stamp stock is 21% of the stamp by weight. For the same size stamp affixed to a #10 envelope with an approximate weight of 5.25 g (0.01 lb), the adhesive amounts to 0.3% by weight of the stamped envelope. Lastly, based upon earlier work by the USPS with the linerless coil stamp at FPL, it was found that a suitable level of adhesive for evaluation purposes was 5% stamp stock, using copy paper as the remaining 95% of the dry weight. A third configuration, Configuration B, consisting of 1.5% stamp stock and simulating the 0.3% adhesive level of a stamped envelope, is also included in the protocol but is an optional level.

Based upon the information provided by the recycling mills on current pulping practices and in an effort to be consistent with the equipment and processes used at FPL, laboratory pulping of the feed stock is carried out using a 0.45-kg (1-lb), high-consistency laboratory pulper (Model 450H, Adirondack Machine). Using this pulper has provided a laboratory-scale means of simulating the high-consistency pulping practiced at the mills and at FPL in the pilot protocol. Furthermore, using a sample size of 360 g (0.72 lb) (ovendried) provides for a sufficient quantity of material from which to sample, especially if the level of contamination is small and extra handsheets are required for a thorough evaluation. In addition, using this pulper provides an indication of the recycling behavior of the adhesives without requiring a large quantity of material. Lastly, the pulper has a variable speed control to allow wetting of the paper at low speed and pulping at higher speeds.

The pulping conditions for recycling in both the laboratory- and pilot-scale protocols were established to include a 15% consistency feed run between 43.3°C and 60°C (110°F and 140°F), typical for recycling MOW. Adhesives evaluated at both STR and FPL at various temperatures in this range demonstrated the temperature sensitivity of the adhesive in the recycling process. Small differences in pulping temperature were found to greatly affect the size of the adhesive particles formed. At the higher temperatures, the adhesives fragmented into many small particles and were more deformable, reducing the adhesive removal ability in subsequent screening steps. A survey of recycling practices by FPL indicated that recycling with furnishes containing stickies is most commonly done between

46°C and 49°C (115°F to 120°F) for efficient removal of adhesive contaminates from the pulp in the screening stages. Therefore, the temperature of the laboratory and pilot protocols was set to 46°C (115°F).

Prior to pulping, the adhesive is stained with Morplas Blue dye. The dyed adhesive stock is laminated to copy paper and all the feed material is shredded into 6.35-mm (1/4-in.) strips that are 27.9 cm (11 in.) long. Tests were conducted to study if it is possible to use a larger paper strip in the pulper so that a closer approximation to the mill situation can be simulated. In the first case, sheets of 21.6- by 27.9-cm (8.5- by 11-in.) copy paper were cut into four sections (10.8 by 76.8 cm (4.25 by 5.5 in.)); 360 g (0.72 lb) of the cut paper were laid flat on the bottom of the pulper; 2040 g (4.08 lb) of 48.9°C (120°F) hot water was then added to the pulper. The pulper speed was started at 150 rpm, gradually increased to 600 rpm. The paper was not completely wetted in this experiment. Also, the paper rotated with the pulper blades at roughly the same speed and no mixing occurred. As the speed increased to 450 rpm, water began to splash out of the pulper.

In another experiment, with the same conditions as previously stated, the soaking time was studied. The paper was laid flat in the bottom of a plastic bucket and 2040 g (4.08 lb) of hot water was added. The paper was soaked for an extended time and examined every 10 min for the first 30 min and every 30 min thereafter. The paper did not wet out after 2 h. Separation of each sheet *is* required to wet the paper completely, which is impossible for any practical purpose. In conclusion, it is very difficult and impractical to use a large-size paper in the pulper. Shredding paper to long strips (6.35 mm by 27.9 cm (1/4 in. by 11 in.)) seems to be practical for the purpose of laboratory repulping studies.

Another set of studies was conducted to determine the possibility of pulping starting at 600 rpm rather than at a lower speed and ramping up to 600 rpm, just like the procedure used in the recycling protocol. Shredded paper was presoaked and transferred to the pulper. An effort was made to fluff or loosen the paper strips as much as possible. The pulper was started at 600 rpm directly and continued for 8 min. Splashing out of the pulper occurred. As a result, it was decided that a ramping stage is needed at the onset of pulping. The final protocol is presoaking the shredded paper with 43.3°C (110°F), pH 10 water, transferring this paper to the pulper, and ramping up to 600 rpm within 2 min.

This practice reduced the time required to ramp the speed from 150 rpm to the final speed of 600 rpm. A total pulping time of 8 min was found adequate to fully defiber the copy paper-adhesive blend.

The next step in the protocol is a two-part screening process in which the diluted pulp is passed through a 0.3-mm (12 cut) Valley flat screen followed by passing the accepts through a 0.15-mm (6 cut) screen, This process is used to simulate the performance of the two pressure screens used at the recycling mills. The pulp passing through the Valley screen is collected in a sieve basket after each screening. Handsheets are made from the initial pulp (10 sheets), after 0.3-mm (12 cut) screening (15 sheets), and after 0.15-mm (6 cut) screening (15 sheets). The handsheets are white and the adhesives particles show up as blue. The amount of adhesive present in the handsheet is estimated by measuring the area and number of blue adhesive particles using image analysis.

The procedure used to evaluate the handsheets is detailed in the USPS image analysis protocol that was prepared jointly by STR and FPL and is presented in Appendix B. For laboratory handsheets made from pulp containing adhesive that was prestained dark blue against a white paper background, the samples are scanned at a threshold setting of 140 gray scale value (gsv). The selection of this level was based upon several studies, two of which are discussed in the following section.

In the absence of a calibration standard, 15.24-cm-(6-in.-) diameter sheets of copy paper having 70 black dots of varying size (similar to the sizes on the TAPPI T437 Dirt Card) printed on them were prepared and scanned using several different threshold settings. The blue prestained adhesive in handsheets is often a combination of light and dark blue specks; therefore, separate sheets with light blue and dark blue dots were also used. The lighter specks, although visible to the eye, will not be detected if the threshold level is not set high enough. The setting where the measured count and area closely matched that of the printed particles and calculated area was at 140 gsv.

Another strategy was to scan handsheets made from experimental recycling trials at several threshold levels and compare the area with that determined by TAPPI T437 procedures using the TAPPI Dirt Card. This run resulted in very low contaminants, after Valley flat screening with a 0.15-mm (6 cut) slotted plate, in the handsheets, making possible a practical use of the dirt estimator card. The results

Suggest that the threshold level of 140 gsv was the best setting for the prestained adhesive. This level was adopted for all future analysis of handsheets for the Optomax image analysis system,

The laboratory recycling protocol is evolving with time based upon studies continually being run to better understand and refine. the various steps in the process and on information provided by members of the recycling industry. Two studies are presented in the following sections to demonstrate some of the findings.

Image Analysis Reproducibility and Reliability Studies

Three separate studies were run to get an indication of the reproducibility and reliability (R&R) of the image analysis measurement system. The studies set out to analyze the variation in the measurements due to the image analysis system and the variation of the measurements due to the instrument operator. The total variation as a result of both the instrument and the operator is referred to as R&R (2). The results of this work are presented in terms of the interval of the measurement; that is, parts per million of adhesive contaminates in handsheets, which represents the estimate of the spread that contains 95% (4 sigma) of the measurement variation due to both instrument and operator. Low values of parts per million representing the R&R spread, denoting low operator and equipment variation, are desirable.

All handsheets used in the analysis were made from pulp after 0.15-mm (6 cut) screening using the USPS laboratory recycling protocol and TAPPI T205 and using different laboratory adhesive recycling trials. The experiments are presented in the following section.

Concluding Remarks

A laboratory recycling protocol has been developed to evaluate both pre- and postconsumer PSA stamp products. The protocol is modeled after mill conditions used for recycling MOW into fine-grade printing and writing papers. Preliminary studies show that the temperature of pulping has an effect on the standard adhesive particle size formation. Pulping at an elevated temperature increases the number of small particles, not all of which can be removed by the 0.15-mm (6 cut) screen. For example, at pulping of 40°C (104°F), larger particles are formed; at 61.7°C (143°F), many more and smaller particles are formed. The use of a lower temperature facilitates removal efficiency of adhesive particles by screening. More smaller adhesive particles are formed in pulp containing release liner

(Configuration C) than without liner (Configuration A). Reducing the stamp level to 1.5% reduces the number of small particles formed in pulping, but the 0.15-mm (6 cut) screen can mostly remove them. At feed levels that might normally be encountered in a mill, the standard stamp laminate material resulting in handsheets from the accepts after screening has very low parts per million. Because of the sensitivity of adhesive removal to pulping temperature, the temperature must be closely controlled.

Pilot-Scale Protocol Development

Removing contaminants from recovered paper pulps is one of the biggest technical barriers to paper recycling (4). Contaminants from adhesives are an undesirable recovered paper component that comes from pitch, ink, plastic films, converting aids, paper coatings, and adhesives. Adhesives are either the hot melt type or the pressure sensitive type. Pressure sensitive adhesives come from products such as labels, tapes, and some postal materials. There are several methods of quantifying contaminants from adhesives, but there is no agreement on a standard method. Despite the advances made during the last few years, contaminants from adhesives, called stickies by papermakers because they stick to paper machine felts and wires, are a major problem during both the processing of recovered paper and the paper-making operation. Closing the water loop by recycling water within mills and shifting to an alkaline-based paper-making process made the problem even more difficult.

The USPS, in conjunction with FPL, STR, and paper industry representatives, organized several national PSA meetings to incorporate the input of the adhesive manufacturing industry and the paper manufacturing industry. The issues included defining the problem, communication, education, source of quality control, recyclability standards for PSAs, paper industry perspective, end-product specifications, and the role of technology. The meetings concluded that problems caused by stickies are best resolved through the combined efforts of the adhesive industry, the paper industry, and the process equipment manufacturers. Based on the criteria set by the paper industry, the adhesive industry is striving to formulate new PSAs that are mostly removable in the screening step. The latest meeting was held at FPL to discuss and decide on the image analysis and pilot plant testing protocols needed to evaluate the newly produced adhesives on the pilot scale. The team agreed that due to a lack of information on the removal efficiencies during recycling unit operations, there is a need for a standard testing method and a need to develop a reasonable method combining pilot testing and image analysis.

As a result of these combined efforts, a pilot-scale separation sequence was developed to assess the removal of adhesives from a feed stock containing PSA. This pilot-scale testing protocol, simulating a typical recycling operation, included high-consistency pulping, coarse and fine pressure screening, forward and reverse cleaning, washing, and flotation deinking. An optical protocol using image analysis was developed to measure residual adhesive. Handsheets made from pulp accept samples taken after each unit operation and on paper samples from the paper machine runs were analyzed by this protocol. The recycling protocol simulating typical recycling operations and using both preconsumer, postconsumer, and control stocks was further refined to reflect optimum operating conditions, such as temperature, consistency, screen size, and repulping energy.

Methods

This section discusses the development of the pilot-scale testing protocol and includes information on the control trials, operating conditions, system cleaning, and testing. The protocol and a flow diagram are shown in Appendix C.

Pilot-Scale Testing Protocol Development

As mentioned, the pilot-scale testing protocol was developed as a joint effort by the USPS, FPL, STR, and various industries representing adhesive manufacturers, paper-makers, stamp paper suppliers, and recyclers. The unit operations were high-consistency pulping (14% consistency, 43°C (109.4°F), 20 min), two-stage (0.3 and 0.15 mm) slotted screening, forward cleaning, two-stage reverse cleaning, flotation, washing, and pressing. A final pulp resulting in a dirt count of 25 ppm or less was set aside for an experimental paper machine run. From those final pulps with a dirt count >25 ppm, a sample was subjected to the additional cleaning operations of dispersion followed by flotation and washing to determine whether the residual adhesive would break down into smaller pieces and be removed by the additional flotation stage.

Sample Contents, Preconsumer, and Postconsumer Simulation

Fourteen stamp stocks containing experimental adhesives were evaluated, with each trial using 90% copy paper plus 10% stamp stock. For the preconsumer trials, the 10% stamp stock was weighed out as received, including the release paper. For the postconsumer trial, the same amount of stamp stock was weighed out but the release liner was removed, the stamp paper was affixed to copy paper. and an extra amount of copy paper equal to the weight of the release liner was added. This 5% stamp stock represents a much greater amount than expected in a typical printing and writing recovered paper stock.

Benchmark and Control Trials

A series of control trials were conducted to provide a reference for assessing the effectiveness and repeatability of the cleaning and screening operations. The four control runs included the following:

- 100% copy paper to determine the best level of cleaning achievable.
- 90% copy paper plus 10% stamp face paper to simulate the best cleaning level without adhesive.
- 90% copy paper plus 10% conventional PSA linered stamp stock to provide a basis of comparison for the experimental adhesives and representing a preconsumer blend.
- 95% copy paper plus 5% lineriess PSA printed stamp stock to provide information on how this new construction would compare with the standard linered stamp, especially the effect of the presence of ink.

Operating Conditions

All operating conditions used for the protocol were to be typical operating conditions. Three temperatures (43°C, 48.5°C, and 60°C (109.4°F, 119.3°F, and 140°F)) were used for pulping during benchmarking trials. A lower temperature was selected for use in the protocol because it gives a higher pressure screening efficiency and represents a typical operating temperature. Pulping temperature can vary from one mill to another, depending on a particular water treatment strategy including the extent of the water loop closure and the integration of the operation. The 14% consistency and 20-min pulping were based on visual observation and pulping energy measurements.

The following changes were made to the Phase IV protocol:

1. Change fine screening basket from a 0.15-mm to a 0.1 -mm (6 to 4 cut) screen to improve screening efficiency and better simulate typical recycling operation
2. Add second stage of forward cleaning to better remove high-density PSAs and be similar to the two-stage reverse cleaning for low-density removal
3. Examine and change surfactant addition point

4. Improve yield by
   - passing pressure screen rejects through a 0.1 -mm (4 cut) flat screen with the accept from flat screen recycled back to the primary system,
   - Installing secondary fiber recovery system for forward cleaners, and
   - Changing sidehill screen size from 0.21 to 0.149 mm (70 to 100 mesh).

System Cleaning

For stock preparation, after each trial, the system was thoroughly flushed with hot water, followed by scrubbing with clean copy paper. Copy paper was then pulped and passed through the system as an additional scrubbing. With a sample taken after sidehilling, 10 handsheets were made, dyed, and checked for residual dirt or adhesive particles. If dirt count exceeded 15 ppm, then an additional scrubbing with copy paper was made. Baskets from the pressure screen were manually cleaned of any adhering adhesive. If the adhesive was more than a trace, the removed adhesive was dried and weighed. The weight was included as part of the rejects for the respective stage. The baskets were then cleaned with a pressure washer plus a manual scrubbing with an organic solvent to remove all remaining traces of adhesive. For the paper machine, the clean copy paper stock from the final stock preparation system cleaning sequence was used to thoroughly clean the paper machine. Samples from the reel were checked for residual dirt. A dirt count of >15 ppm off the reel indicated that an additional cleaning sequence needed to be made. Clean copy paper was pulped and used for additional cleanings.

Testing

Each material was evaluated in two trials, one simulating postconsumer loading (no release liner present) and the other representing a preconsumer loading (release liner included). During each trial, an accepts pulp sample was collected at each unit operation outflow. Handsheets made from these samples were dyed and read optically for dirt count; that is, the adhesives content. Weight, consistency, and flow rate were measured and calculated as necessary to provide a mass balance and yield information for each trial. Energy input to the pulper was also collected for each trial, and, if pulp was cleaned sufficiently, paper machine runs were made. A PIRA deposition apparatus was used to conduct tests on all final pulps. Biochemical oxygen demand (BOD) on the wastewater from washing and mineral metal analysis of the fresh water used and the wastewater from washing were collected. Visual observation on the behavior of each experimental adhesive during recycling was noted at each stage. Equipment, especially the pressure screens, was visually observed after each run to examine if adhesive deposition was a problem or not. Blank runs were made with clean pulp until the dirt count of the final pulp was <10 ppm.

Venditte and others (5) at North Carolina State University examined two methods to determine stickies content of pulp containing PSA and found correlation between image analysis results of dyed handsheets and deposition tests using the PIRA deposition tester.

Discussion

The behavior of both control (standard) and one experimental adhesive is discussed and analyzed for each unit operation of the protocol. The results of the speckcheck analysis are shown in Table 1.

Table 1. Speckcheck analysis (parts per million basis)

Trial		Contaminant level (ppm)							
		Initial pulped slurry	0.30-mm pressure screen accepts	0.15-mm pressure screen accepts	Forward cleaner accepts	Through-flow cleaner accepts (-pass 1 (-pass 2))	LaMort flotation accepts	Sidehill washing accepts	Pressing/ shredding accepts
Controls									
127	Copy paper only	1.21	3.17	2.91	0.86	1.85 (2.12)	1.34	5.82	9.78
128	Face paper only	2.09	1.66	2.01	1.31	2.06 (1.27)	2.11	1.59	2.17
107	Linerless stamp stock (43°C)	913	54	9.4	6.4	10 (1.6)	2.1	0.6	5.2
106	Linerless stamp stock (48.5°C)	1,506	63	21	31	52 6.7	2.4	2.7	1.6
110	Linerless stamp stock (60°C)	1,734	338	101	49	26 23	12	18	10
109	Standard PSA stamp stock w/o release liner	2,093	601	42	31	20 21	13	10	19
108	Standard PSA stamp stock with release liner	3,002	999	112	94	79 84	20	26	21
Experimental adhesives									
131	Adhesive A w/o release liner	2,120	424	35.3	13.4	22.44 22.68	0.51	7.51	7.45
132	Adhesive A with release liner	2,622	85	41.6	24.3	57.93 13.13	13.99	11.84	12.24

Pulping and Screening

The objective of the pulping stage is to fiberized the paper and keep the contaminants large. Generally, this is made easier if the conditions are right to agglomerate adhesives without blinding the screen.

Preliminary trials indicated that a 20-min pulping time at pH 10 would be satisfactory for both the copy paper and the stamp face paper. A shorter time left tags, which are small pieces of paper. Longer pulping times tended to break down the contaminants into smaller particles plus waste energy and increase batch turnaround time. Energy measurements were made to make sure that 20 min was the optimum time needed to liberate the fibers and keep the contaminants large and screenable.

The separation of fibers from the contaminants is desirable for high-recovery pulp yields. Having a few fibers attached to a contaminant may be desirable if their presence enhances the removal of the particle in a unit operation, such as screening where bulk is important. Their presence can be detrimental if the fibers shift the effective density towards 1.0, thereby nullifying the effectiveness of centrifugal cleaning. Entanglement of the attached fibers with free-flowing fibers in a forward cleaner will also impede the movement of the particles in the forward cleaner.

In this study, little evidence of fiber attachment was found on any adhesive. Adhesive particles rejected by the pressure screen and retained by the flat screen all seemed to be free of fiber. The fiber observed through a 30 × lighted magnifier appeared to be sandwiched between adhesive particles as they were wadded together during collection. The cleanliness of the adhesive seemed to depend on how thorough a wash the adhesive was given on the flat screen. Tags were retained by the flat screen and were prominent in the rejected adhesive, but individual fibers were generally not retained and relatively few showed up in the adhesive. Had the adhesive maintained the paper-adhesive bond during the pulping, there should have been adhesive particles that were virtually coated with fiber. This was not evident for any of the adhesives.

The presence of fibrous tags increased the dried weight of the collected adhesive, giving in some cases a false high value of rejects and a false sense of screening effectiveness. In the same manner, there are particles of adhesive that pass through the 0.2-mm (8 cut) flat screen, an action that lowers the weight of the rejected adhesive, thereby decreasing the apparent screening efficiency. For these reasons, it was determined that the decrease in dirt count for the screening accepts relative to the screening feed is a better indicator of the true screening efficiency and yield in general.

For the preconsumer trials where release liner was included, the collected adhesive generally showed a smaller grain structure than for the postconsumer trials where the release liner was absent. As the paper disintegrated into fiber during pulping, if you assume that an adhesive in both cases is (i) released into the slurry having the same particulate size and form, then (ii) proceeds to reagglomerate into larger particle groupings, the finer structure of the preconsumer adhesives would imply that one or more components of the release coating (for example, silicon or clay) impedes the reagglomeration. If this scenario is correct, the adhesive groupings should be three-dimensional, implying that for the same amount of adhesive, the summation of the cross-sectional areas of the larger groups should be less than the summation of the areas of the smaller groups. This implies that the dirt count parts per million should be greater for the smaller particle grouping than for the larger particle grouping. That is, the handsheets from the pulper (before screening) for the trials with release liner should have a higher parts per million than the same handsheets for the no-liner trials. This was the case for all but one adhesive used in this study.

Standard Adhesives

Control adhesives used for benchmarking are the linerless and the standard PSA stamp stock with and without liner. The linerless stamp stock results are shown in Table 1. Trial 107 (linerless at 43°C (109.4°F)) shows a very high pressure screening efficiency, from initial dirt count of 913 ppm to about 10 ppm after two-stage screening, and a very clean final pulp of about 2 ppm after flotation. Results from standard PSA construction (Trials 108 and 109) are also shown in Table 1, After two stages of pressure screening, the dirt count for the standard stock without liner (postconsumer simulation) went from initial 2,093 to 42 ppm and gave final pulp, after flotation, with 13 ppm. The trial with liner gave much lower pressure screening efficiency, apparently due to some material in the liner (clay, silicon), causing some pacification. One objective of this project was to evaluate the new experimental adhesives and determine if they screen better and are less problematic during processing. One experimental adhesive called Adhesive A is analyzed in this paper.

Experimental Adhesive

Experimental Adhesive A was primarily removed by the 0.30-mm screen with only a few fiber tags present. The adhesive dried to a gritty consistency, free of fiber except for the tags. Examination of the dried adhesive using a 30 × magnifier showed the adhesive particles to be glass-like beadlets. A similar examination for the rejects showed a similar appearance. Interestingly, the magnified view also revealed the presence of many tiny colored particles in the linered trial pulp. It was assumed that these were ink particles, indicating the presence of recycled fiber in the base sheet of the release liner. These particles were small enough to have been washed through the slots of the flat screen. That they were still present on the adhesive indicated an affinity for the ink by the adhesive.

Adhesive removal efficiencies more than 100%, based on weight of dried rejects from the flat screen, are dubious. These negative removals reflect the presence of fibrous tags in the rejects. A removal efficiency based on residual adhesive in the accept streams as a ratio of the feed concentration given as parts per million is a more reliable indicator. For Adhesive A, the removal efficiencies using this technique were 80.0% and 98.330% for the 0.30- and 0.15-mm (12 and 6 cut) screens. respectfully, for the trial without liner, and 67.43% and 98.41%, respectively, for the trial with liner. Actual image analysis values are presented in Table 1. The early removal of most adhesive, 90% to 95% via the screens, is important in that the smaller amount of residual adhesive left in the pulp has a higher probability of responding to the removal action of the subsequent unit operations. The response of Adhesive A is excellent, despite the lower initial removal in the 0.3-mm (12 cut) screen when the liner was included. Apparently, the basic adhesive particle is fairly massive and rejected by the 0.15-mm (6 cut) screen even if it does not reagglomerate after pulping.

Forward Cleaning

Adhesive A was apparently a denser adhesive (a sinker) as it responded to the forward cleaning. Without liner, the pulp decreased from 35.3 to 13.38 ppm (62% removal), and with liner, it decreased from 41.6 to 24.3 ppm (42% removal). The moderate decrease in dirt count indicates a density of just over 1.0. Generally, there is about a doubling of the particle count for the linered trial versus the no-liner trial after pulping, indicating a retardation of the agglomeration of the adhesive particles in the linered pulp. For Adhesive A, there was only a small dirt count increase (2,120 to 2,622 ppm). The retardation may be due to a component of the release liner. For Adhesive A, with its larger particle size, there should be a much lower surface area to adhesive mass. If a coating of the particle is what retards the agglomeration, then clay may be the coating component. There might be sufficient clay in the copy paper to coat the large particles and any additional amount coming from the release coating would not have much of an effect; that is, the particles would not agglomerate even if the release backing is excluded. The very gritty appearance of the adhesive for both trials indicates this. For the forward cleaner, the small additional coating of clay may, for a nearly neutral density adhesive, be just enough to nearly neutralize the cleaner action.

Reverse Cleaning

Since Adhesive A is apparently a denser adhesive, it should not respond to the action of the reverse cleaner, which is designed to remove low-density particulates. The dirt counts for this trial reflect this anticipated result. For the linered pulp, the results were mixed but imply a possibly slight increase in dirt count. If the adhesive is indeed coated with clay, the slight shift in density could explain the cleaner action.

Flotation

Adhesive A seemed to respond to flotation in the trial without liner, decreasing the dirt count to 0.51 ppm. There was virtually no change in the trial with liner. Both results must be taken cautiously because with large particles, the dirt count is accounted for by only a few particles and a numerical change of just a few particles can swing the dirt counts significantly. For the no-liner trial, this numerical change was from 43 to 3 particles, these being in the 0.02 to 0.03 mm^2 (31 to 47 × 10^{-6} in^2) range. For the liner pulp, the change was from 25 to 11 particles in a size range up to 0.8 mm^2 (0.00124 in^2). These results imply that the coating might be interfering with the flotation action.

Washing, Pressing, and Shredding

Washing removes very small particulates: clay, fiber fines, etc. Adhesive floaters tend to remain with the pulp thereby becoming more concentrated as material is washed away. Adhesive sinkers are removed only if they are very small particles. Typically, sidehill washing shows an increase in dirt count. Adhesive A seems to behave in this manner, increasing in concentration for the no-liner pulp and maintaining its level for the linered trial. Again, the few particles present can cause wide swings in the dirt count with a change in the number of only a few particles. Pressing and shredding of the cleaned pulp should have no effect on dirt count. For both trials, the final pulps are about the same as the sidehill.

System Cleaning

Cleanup of the equipment was not a problem with Experimental Adhesive A. Adhesive residue on the tanks washed off easily with 85°C (185°F) hot water. Dirt counts of the cleanup (flushing) pulp coming off the sidehill screen were about 3 ppm for both postconsumer and preconsumer trials.

Concluding Remarks

Adhesive A appears to be a desirable adhesive for recycling. The majority of the particles were removed by the 0.3-mm (12 cut) slots, with virtually all of the rest being rejected by the 0.15-mm (6 cut) slots. The basic particle seems to be very large such that the normally encountered failure to agglomerate in the presence of the release liner is no problem. If there is a problem, it is the density apparently being too close to 1.0 so that the cleaners are neutralized and the flotation is too sensitive to slight shifts in density as a result of possible coating action of contaminants. A slight shift to a lighter density might be recommended, providing the change does not affect the size distribution of the particles.

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