PULP EXTRUSION AT ULTRA-HIGH CONSISTENCIES: SELECTION OF WATERSOLUBLE POLYMERS FOR PROCESS OPTIMIZATION

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ABSTRACT

Pulp extrusion at ultra-high consistencies (20% to 40% solids) is a new process developed at USDA Forest Service, Forest Products Laboratory (FPL) to convert recovered papers, wastepaper, and papermill residuals into solid sheets or profiles for compression molding. This process requires adding a water-soluble polymer (WSP) to alter the rheological properties of the pulp and generate a paste that can be extruded. The variety of fibrous raw materials can have a significant impact on the efficiency of a WSP to alter viscosity. Therefore, an appropriate WSP must be selected that will rapidly hydrate and adhere to fiber surfaces, allowing flocs to disperse in the shear-intensive environment of an extruder. Also, temperature-dependent viscosity changes are important to control elongational flow for fiber alignment in the extrudate and to enhance the consolidation properties. This study presents a methodology for determining the rheological properties of fiber pastes. Selected WSP (natural and modified gums, cellulose derivatives, and gelatin) were added to a clean newsprint pulp and processed in a modified bowlmixer rheometer. The temperature of the bowlmixer was controlled and ramped between 20°C and 100°C to observe temperature-dependent viscosity changes. Various cations were also added to induce thenno-reversible viscosity changes. A blend of sodium carboxy-methylcellulose and hydroxypropyl-methylcellulose resulted in the best overall rheological properties.

INTRODUCTION

Pulp extrusion was originally conceived as a process to convert underutilized wastepapers and papermill residuals into products similar to millwork or hardboard [1]. Extrusion of wood-fiber/polymer composites is common practice. But the wood component is typically used only as a filler, because it is difficult to generate a dry fiber component without severely damaging the fiber. Our goal was to develop an extrusion process that could accommodate a feedstock of pulp at ultra-high consistency (20% to 40% solids). Conventional pulping techniques could then be used to produce a crumb pulp that doesn't need to be completely disintegrated or "cleaned" and yet retains fiber integrity.

Several factors must be considered to develop a successful pulp extrusion process [2–5]. Among these are wastepaper characteristics (fiber length, fiber coarseness, fiber flexibility, residual lignin, filler type or content, and contaminant levels), extruder configuration (screw configuration, barrel temperature profile, and die design), and consolidation technique (air-drying or restraint-drying). However, the most important consideration is the selection of a water-soluble polymer (WSP) to alter pulp viscosity and develop a fiber paste that can be extruded.

Based on previous experience in selecting a WSP for pulp extrusion, it has been determined that there are six specific polymer requirements that must be considered. The first requirement is rapid hydration (within 10 seconds), preferably at ambient temperature. This is critical because it makes it possible to simultaneously add the polymer to the pulp as it is being fed into the extruder without a preblending step. Aggressive mixing elements can then be arranged in the feed zone of the extruder to quickly disperse the polymer and generate a paste. The second requirement is that the WSP have a high molecular weight (>500,000). This is also critical because only the very high molecular weight WSP give dramatic viscosity reductions. The third requirement is an affinity for cellulose. So far, the most effective and efficient WSP are cellulose derivatives. The fourth requirement is low shear sensitivity. The screw configuration best suited for pulp extrusion includes several intense kneading zones to completely disintegrate and disperse the fiber flocs and produce a homogeneous paste. The WSP must be tolerant of high shear stresses developed in these zones. The fifth requirement is that the polymer be nonhazardous. The sixth requirement

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is that the WSP be thermo-reversible (that is, hydration is temperature dependent and reversible). The significance of this requirement is that if a WSP can be identified that will dehydrate at an elevated temperature, then it will be much easier to densify and dry the extrudate. This thermo-reversibility requirement is the subject of this paper.

Of the numerous WSP previously investigated, none has matched the effectiveness of sodium carboxymethyl-cellulose (CMC) in developing a fiber paste suitable for extrusion. However, CMC is only moderately temperature sensitive and not thermo-reversible. Thus, a search was initiated to find a WSP that met all of the six requirements. A seemingly infinite array of industrial gums is available [6], but only a few are known to be thenno-reversible (for example, gum blends of xanthan and locust bean, borated gels of guar, or cation interactions with carrageenan). It seems, however, that little is known about the mechanism of thermo-reversibility or of potential interactions with cellulose fibers.

EXPERIMENTAL

A modified bowlmixer rheometer is used to measure the rheological properties of fiber pastes (Figs. 1-3). The bowlmixer incorporates a system of two counter-rotating paddles that knead the pulp. The paddle design shown is very similar to the kneading elements used in an extruder. A charge of crumb pulp (usually 40 g) is initially added to the bowl, followed by a charge of WSP. (All WSP charges are based on percentage ovendry weight fiber and are usually in powder form.) An in-line torque sensor measures the torque required to maintain a mixing speed of 200 rpm as the WSP is dispersed. The torque response (apparent viscosity) is continuously recorded. The bowl housing is also surrounded by a water jacket to control the pulp temperature by regulating the flow of cold and hot water. Temperature sensitivity of the WSP is determined by cycling the temperature between hot and cold and observing changes in apparent viscosity as well as the presence of "free" water. The range in apparent viscosities measured with this bowlmixer rheometer has been found to correlate well with the torque response experienced in actual extrusion trials.

Based on published observations and previous experience, several WSP samples were collected for rheological evaluation. These included CMC, hydroxypropyl-methylcellulose (HPMC), xanthan, guar, agar, carrageenan (kappa and iota), carboxymethyl-starch, hydroxypropyl-cellulose, and gelatin. A clean newsprint furnish was selected for these trials. Swatches of newsprint were removed from a stub roll and pulped at 10% consistency. The pulp was then dewatered in a bladder press to 30% solids and disintegrated in a shredder to obtain a crumb pulp (Fig. 4).

RESULTS AND DISCUSSION

The first bowlmixer trial was on the newsprint crumb pulp without WSP added (Fig. 5). This first trial demonstrates the typical protocol followed for all tests. After about 30 seconds, the pulp was slowly added to the mixing chamber (taking about 30 seconds). The mixing continued for another 30 seconds to establish an equilibrium torque response. At this point, the apparent viscosity was quite high and water was continually squeezing out of the flocs, producing large fluctuations in torque readings. At about 120 seconds, the temperature was increased to 60°C with a corresponding decrease in apparent viscosity. At about 240 seconds, the temperature was increased to about 100°C with another decrease in apparent viscosity. The mixing chamber was then cooled, and torque began to increase. In the case of raw pulp, the apparent Viscosity was not reversible because the mixing action had severely damaged the fibers.

The second bowlmixer trial was newsprint with 3% CMC added at about 90 seconds (Fig. 6). At this point, there was a dramatic torque drop that signified rapid hydration of the polymer. A stable torque level of about 1 Nm was achieved as the fiber flocs were dispersed and a homogenous paste was formed. When the temperature was increased to about 100°C, a small decrease in apparent viscosity was observed. Upon cooling to 20°C, the apparent viscosity increased only moderately.

The third bowlmixer trial was similar to the second, but at 240 seconds, 100 mg (0.84%) of Alum was added (Fig. 7). Upon addition of the Alum, there was an immediate increase in apparent viscosity and the paste was somewhat destabilized. However, when the temperature was increased to 100°C (at 500 seconds), a stable paste was again apparent. Upon cooling, the apparent viscosity again increased. The addition of Alum to CMC imparted a small degree of thermo-reversibility. However, if an additional 50 mg (0.42%) was added, the apparent viscosity dramatically increased, free water was apparent, and the CMC would not rehydrate regardless of the temperature.

The fourth trial was newsprint with 3% HPMC added (Fig. 8). Like CMC, there was a dramatic torque drop signifying rapid hydration of the polymer. An equilibrium torque level of about 2 Nm was reached with some instability. For HPMC, this instability is indicative of shear sensitivity. As the temperature was increased, the apparent viscosity slowly decreased and stabilized. However, at about 63°C, a dramatic increase in apparent viscosity was observed. The HPMC quickly dehydrated, and free water was apparent When the temperature was decreased a homogenous paste reformed. This process can be repeated with the same results. Therefore, HPMC meets the criteria for a thermo-reversible WSP.

The fifth trial was newsprint with a blend of 2.6% HPMC and 0.4% CMC (Fig. 9). As expected, a dramatic torque drop was observed and a stable torque level was achieved When the temperature was increased, the apparent viscosity stabilized even more and a uniform paste similar to that of the CMC trial was observed. Once the temperature reached 75°C, however, the apparent viscosity increased and the paste destabilized. Upon further cooling, a homogenous paste reformed. The WSP blend used in this trial can also be considered to be thermo-reversible. However, if the ratio of HPMC to CMC is decreased from 7: 1 to 3: 1, the paste will not dehydrate when heated.

The sixth trial was newsprint with 2.25% guar added (Fig. 10). The torque drop was not dramatic, but once the temperature was elevated, a very nice paste developed. The paste apparent viscosity was only moderately affected by temperature cycling, but when 20 mg (0.27%) of sodium tetra-borate was added, the pulp began to exhibit thermo-reversibility. However, unlike HPMC, the borated guar dehydrated when cold. A similar response was observed when potassium chloride was added to kappa carrageenan (at a carrageenan to KCl ratio of 3.75:1).

The seventh trial was newsprint with 6.75% agar added in thirds about 100 seconds apart (Fig. 11). The agar did not appear to hydrate cold, so the temperature was immediately increased. Once hot, however, the agar seemed to form a nice paste at a torque level of 1 Nm. When the temperature was decreased, the agar began to dehydrate and the , paste destabilized. This affect is repeatable, and the agar is considered to be naturally thenno-reversible.

The eighth trial was newsprint with 4.5% gelatin added in two equal parts (Fig. 12). Like agar, the torque drop was not dramatic and the pulp had to be hot for hydration. But unlike agar, the gelation did not seem to interact with the pulp fibers and a homogenous paste was not observed.

CONCLUSIONS

Pulp extrusion at ultra-high consistencies (20% to 40% solids) is a potentially useful process for converting recovered paper, wastepaper, and papermill residuals into solid sheets or millwork. To produce a uniform extrudate from pulp, a water-soluble polymer (WSP) must be added to alter the viscosity of the pulp and produce a fiber paste. For this to occur, the WSP must meet six very specific requirements: rapid hydration (within 10 seconds), high molecular weight (>500,000),. affinity for cellulose, low shear sensitivity, nonhazardous, and thermo-reversibility. However, these requirements can also be conflicting. For instance, a WSP that hydrates quickly has a strong affinity for water. An excellent fiber paste is generally observed, but the paste is also difficult to densify in a compression mold because it flows easily under pressure. Also, when drying the extrudate, additional energy is required to dehydrate the WSP. A WSP that dehydrates at elevated temperatures is needed.

Several industrial gums meet many of these requirements. Natural gums such as guar, agar, and carrageenan can develop good pastes, but each has its limitations. For instance agar produces a very nice paste and is naturally thermo-reversible, but the reversal occurs at cooler temperatures. Guar also develops a very nice paste but requires the presence of borate ions to become thenno-reversible. Even then, the borated guar is thenno-reversible at cooler temperatures.

Cellulose derivatives seem to the most efficient WSP. Sodium carboxymethyl-cellulose (CMC) seem to produce the best fiber paste and has been used almost exclusively in previous extrusion trials. However, CMC can only be made moderately thermo-reversible by adding small amounts of alum. But again, this reversibility occurs at cooler temperatures. Hydroxypropylmethyl-cellulose (HPMC) also generates a nice fiber paste and has a very dramatic change in apparent viscosity at high temperature. Once a threshold temperature is reached, the HPMC quickly dehydrates and free water is apparent. If the pulp is cooled, a paste is reformed. Unfortunately, the HPMC is

somewhat sensitive to shear stresses. However, this can be overcome by adding a small amount of CMC. Thus, a blend of HPMC and CMC is the most likely candidate for further study and extrusion trials.

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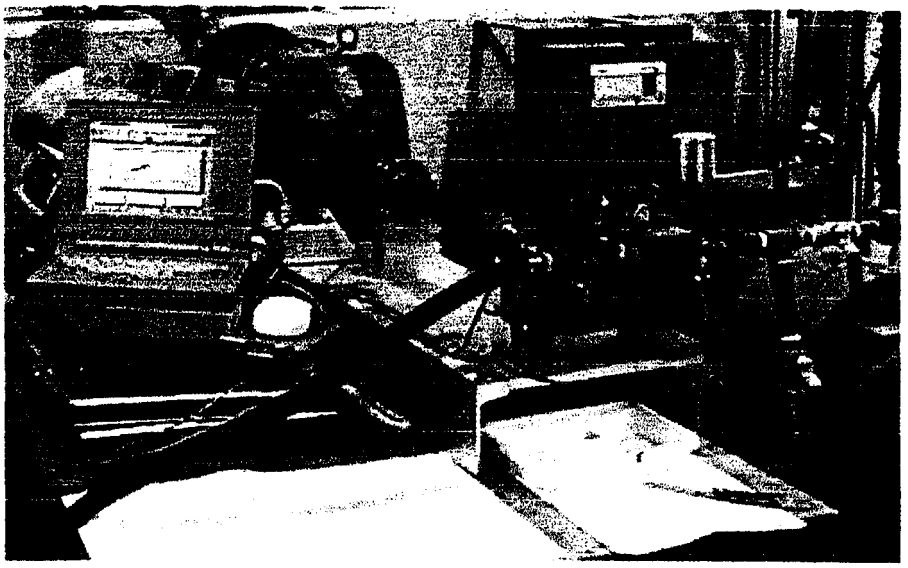

Figure 1. Experimental configuration of bowlmixer rheometer used to measure the rheological properties of fiber pastes.

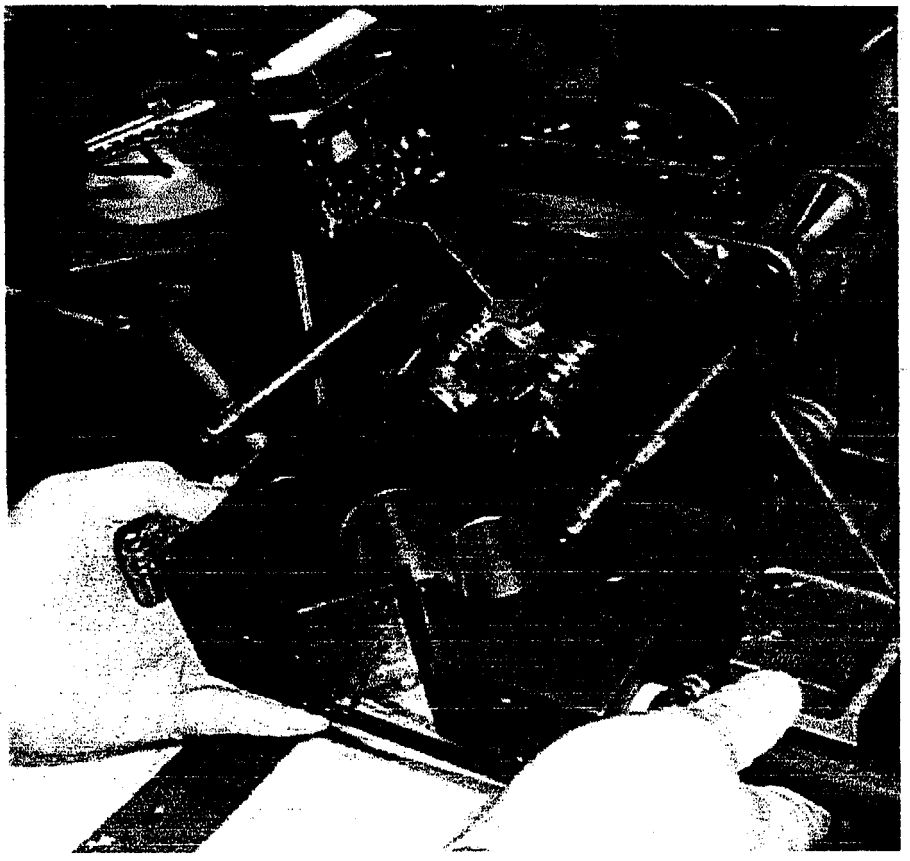

Figure 2. Bowlmixer mixing chamber and paddles. The mixing chamber incorporates a water jacket for heating and cooling the fiber paste.

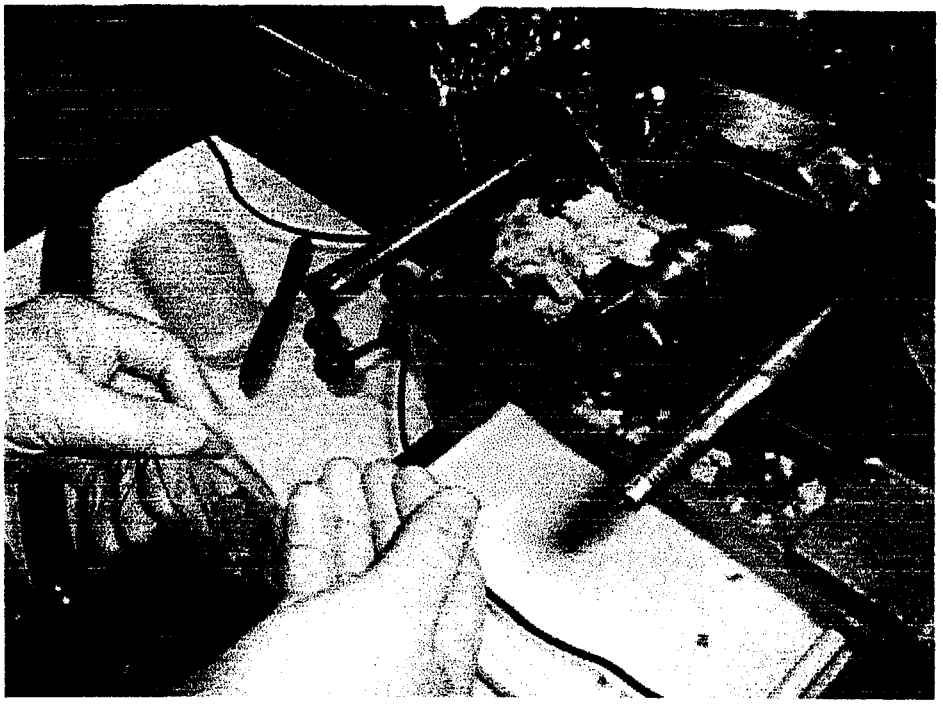

Figure 3. Collecting the fiber paste after a bowlmixer trial.

Figure 4. Samples of crumb pulp before a bowlmixer trial (right) and a fiber paste after mixing (left).

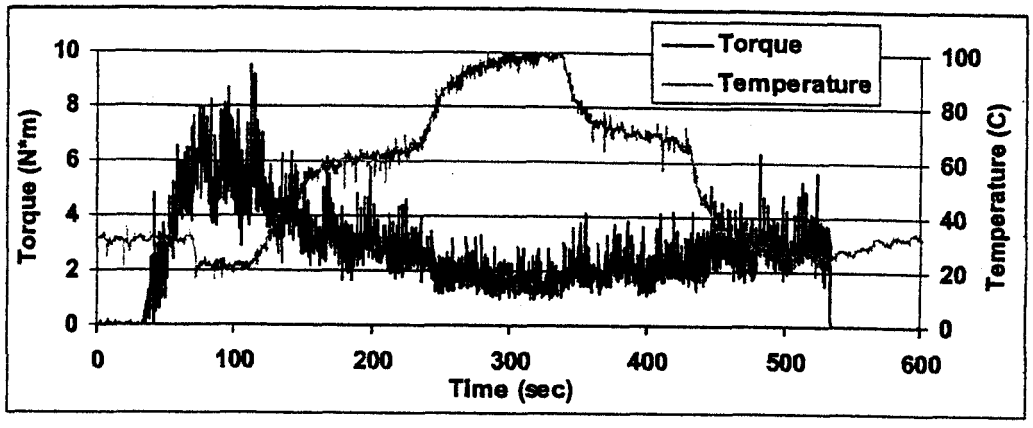

Figure 5. Apparent Viscosity of newsprint crumb pulp as a function of temperature.

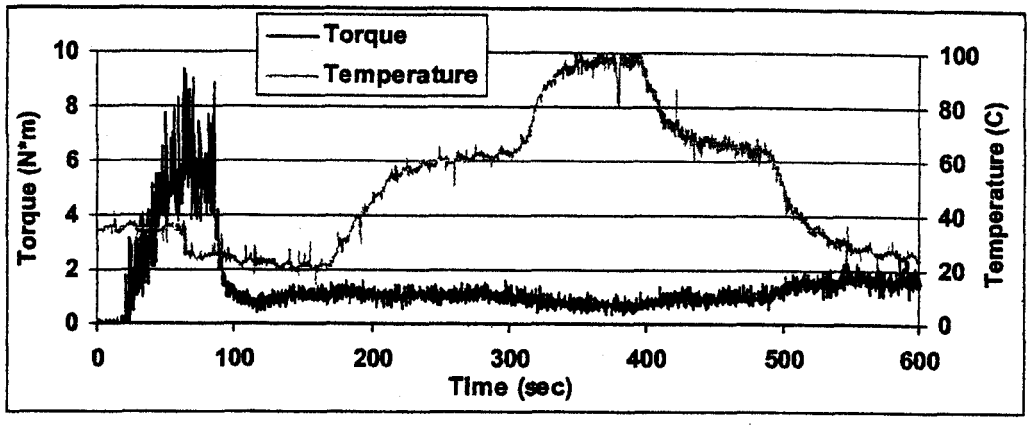

Figure 6. Apparent Viscosity of newsprint + 3% CMC.

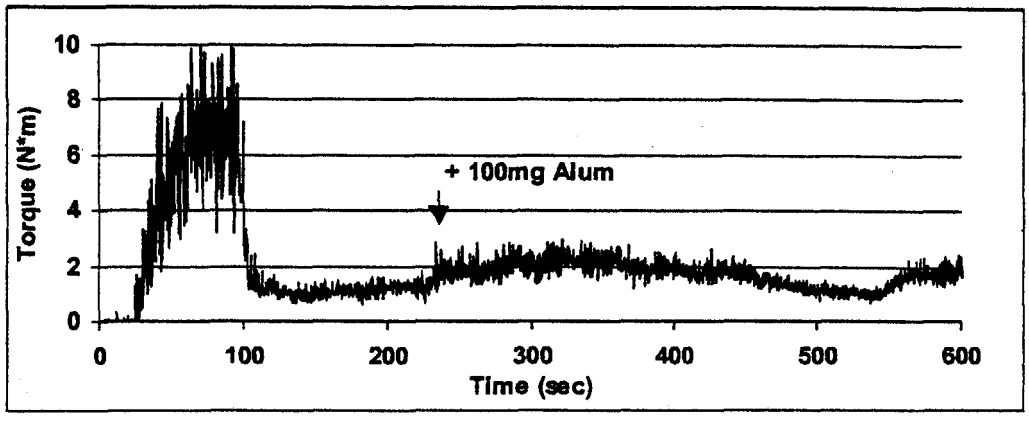

Figure 7. Apparent Viscosity of newsprint + 3% CMC with 100 mg of alum added at 240 seconds. The apparent viscosity change between 400 and 600 seconds was due to a heat cycle (not shown).

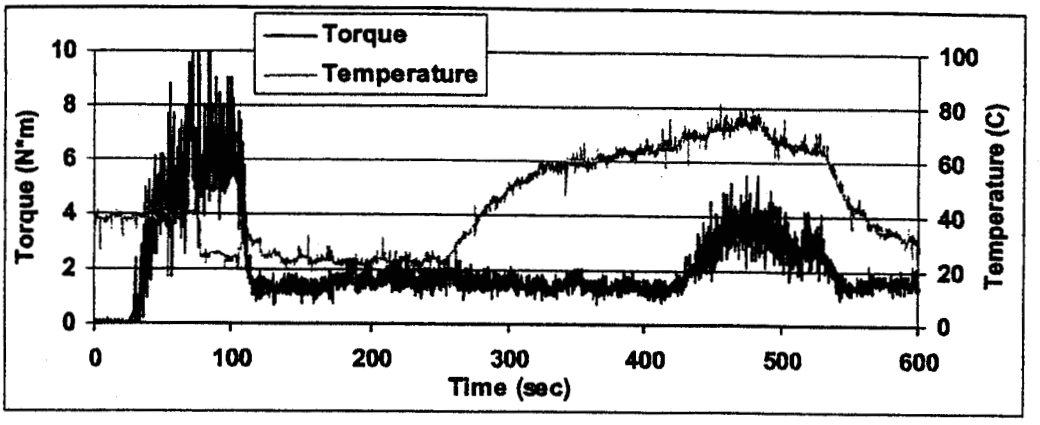

Figure 8. Apparent viscosity of nespring + 3% HPMC. Note the dramatic apparent viscosity reversal at about 63°C

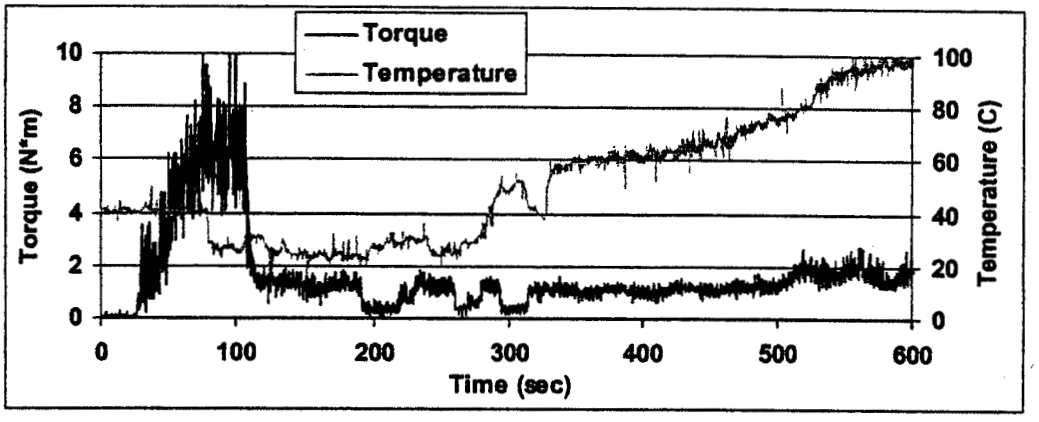

Figure 9. Apparent Viscosity of newsprint + 2.6% HPMC + 0.4% CMC. Note the moderate apparent viscosity reversal at about 75°C.

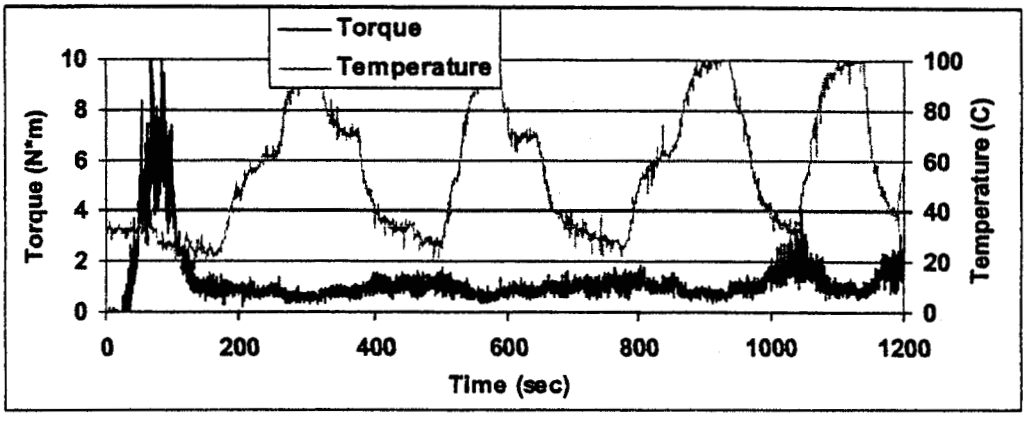

Figure 10. Apparent viscosity of newsprint + 2.25% guar with 10 mg sodium tetraborate at 450 seconds and again at 725 seconds. The paste became thenno-reversible after one complete heat cycle.

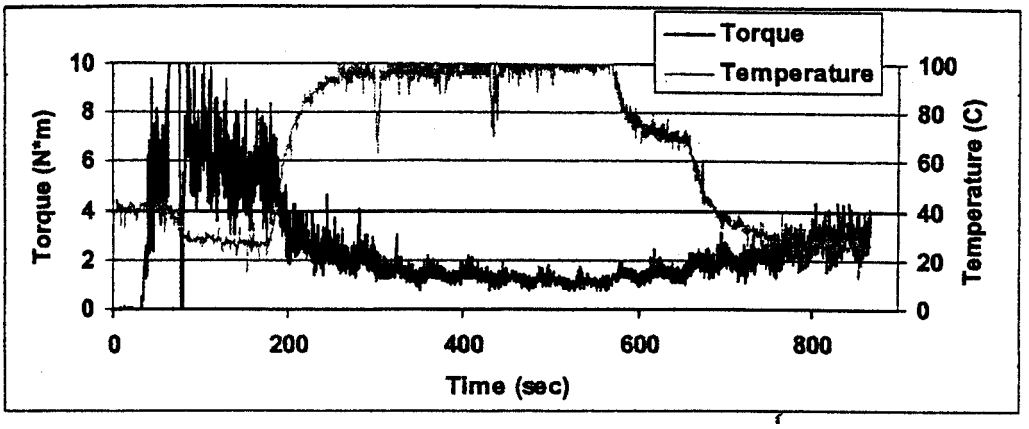

Figure 11. Apparent viscosity of newsprint With 2.25% agar added at 150 seconds, 300 seconds, and 450 seconds. Note the apparent Viscosity reversal upon cooling.

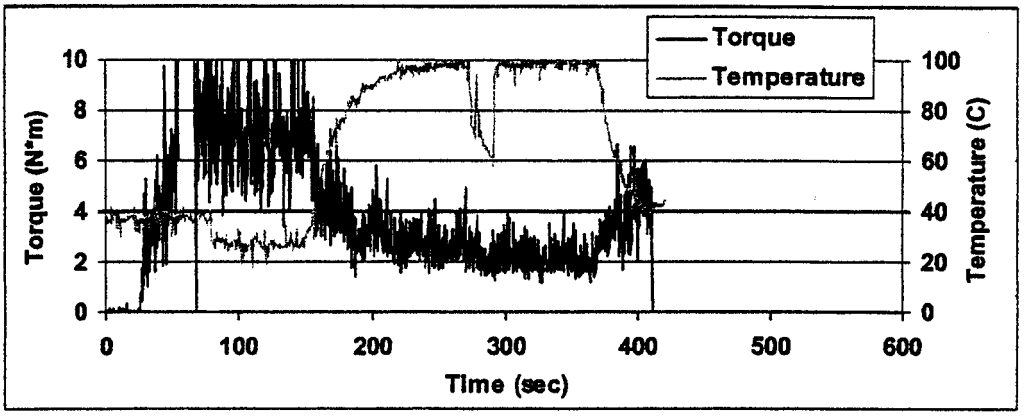

Figure 12. Apparent viscosity of newsprint with 2.25% gelatin added at 150 seconds and again at 275 seconds. Note the apparent viscosity reversal upon cooling.