

THE HALF-LIFE OF BIOLOGICAL KNOTS IN KRAFT PULPING

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ABSTRACT

How many times do you have to cook biological knots and the knotted rejects derived from them in a bleachable-grade kraft mill before they are reduced completely to fibrous material and dissolved organics? And what is the yield of pulp from such material? To find the answers, we experimented with stockpiled knotted rejects and with fresh biological wood knots. With either material, re-cooking demonstrated that knotted rejects continued to become further knotted rejects. Relative to normal wood chips, biological knots cooked far slower and to lower pulp yields; after re-cooking, the pulps became progressively worse in average fibre length and overall strength. Re-cooked knotted rejects from biological knots had a half life of two complete cooks in bleachable-grade kraft pulping. Because mixing biological knots or their knotted rejects descendants with normal chips makes kraft pulping much more heterogeneous and impairs pulp yield and product quality, they should be purged as efficiently as possible from chip furnishes going to digesters, and knotted rejects containing them should go to hog fuel boilers or landfills rather than digesters.

RÉSUMÉ

Combien de fois faut-il cuire les nœuds biologiques et les rejets du trieur de nœuds qui restent dans une usine de pâte kraft blanchissable avant qu'ils soient réduits entièrement en matière fibreuse et en matières organiques dissoutes? Et quel est le rendement en pâte de ces matières? Pour trouver les réponses, nous avons procédé à des essais avec des rejets du trieur de nœuds accumulés et avec des nœuds biologiques de bois vert. Avec l'une ou l'autre des matières, une nouvelle cuisson a démontré que les rejets du trieur de nœuds continuaient de produire d'autres rejets du trieur de nœuds. Par rapport à des copeaux de bois ordinaires, les nœuds biologiques cuisaient beaucoup plus lentement et leur rendement en pâte était inférieur; après une nouvelle cuisson, les pâtes empiraient graduellement en ce qui a trait à la longueur moyenne des fibres et à la résistance globale. Les nœuds biologiques rejetés au trieur de nœuds avaient une demi-vie de deux cuissons complètes pour la mise en pâte kraft blanchissable. Parce que mélanger des nœuds biologiques ou des

descendants des rejets du trieur de nœuds avec des copeaux ordinaires rend la mise en pâte kraft beaucoup plus hétérogène et nuit au rendement de la pâte et à la qualité du produit, ces nœuds devraient être éliminés le plus possible des compositions de fabrication de copeaux alimentées aux lessiveurs, et les rejets du trieur de nœuds qui en contiennent devraient être acheminés aux chaudières brûlant des déchets ligneux ou encore mis en décharge, plutôt que d'être alimentés aux lessiveurs.

INTRODUCTION

In kraft pulping, some wood particles are more resistant to delignification than others. Two kinds are particularly troublesome – overthick wood chips (typically greater than 8 mm), and biological knots (emanating from any branch wood inside a tree's stemwood which was cut into chip-like form and size unintentionally). Figure 1 shows biological wood knots: there is a dense core with a circular cross-section, surrounded by reaction wood of non-uniform dimensions and orientation. Neither kind of particle will completely delignify beyond the fibre liberation point in bleachable-grade kraft pulping, resulting in wood “cores” or knots amidst the fibrous pulp from a digester. All kraft mills have knotting equipment to separate cooked knots from pulp.

But once separated as knotted rejects, what should be done with them? There are three options, more than one of which may be used at a given mill [1]:

1. **Recycle** the knotted rejects back to chip delivery to the digester. This captures their “wood fibre potential” in a yield sense, but it also assumes that they can be pulped successfully the second time around, just like wood chips of proper thickness.
2. **Refine** them into pulp mechanically in a stock refiner, and add this pulp to the main flow of fibrous pulp going forward in the mill. This option is typical in linerboard and unbleached kraft paper mills, where significant amounts of knotted rejects are always being generated because cooking is deliberately terminated at kappa number targets above the fibre liberation point. Usually, the pulp from reject refining joins the linerboard basestock stream.
3. **Remove** the knotted rejects from a mill's fibre line, and send them to a landfill or a power boiler.

In bleachable-grade kraft mills, it is common to use some combination of Options 1 and 3 to deal with knotted rejects; Option 2 risks contaminating the pulp with small bits of darker material which resist further delignification and bleaching, impairing the cleanliness of the final pulp.

Unfortunately, the common terminology in this area of kraft mill operations doesn't properly describe "knots" or "knotter rejects", nor is this stream routinely analyzed for its real contents. The material rejected by knotters usually has several distinct components: uncooked wood cores from overthick chips (which would have been cooked completely to fibrous pulp had the chips been of appropriate thickness), biological knots (from branch wood that is highly resistant to kraft pulping), and non-wood particles such as rocks and pieces of metal, concrete, rubber, and hard plastics. Knotter rejects can be accompanied by significant amounts of fibrous and near-fibrous pulp associated with almost-successful cooking, still clinging to or entangled with the surfaces of the larger rejects due to incomplete separation in pressure knotters.

The delignification of overthick (but otherwise normal) wood chips is strictly a function of thickness, and all chips thinner than about 8 mm will normally be reduced to pulp fibres in bleachable-grade kraft pulping [2,3]. Biological knots, however, present a more serious challenge [4-6]. As wood particles, they are very often overthick because their higher density than the surrounding wood and their circular geometry make them more resistant to being cut into normal chip-like form in chippers. Worse still, they are enriched in lignin content relative to stem wood, further increasing their resistance to delignification.

To minimize the generation of knotter rejects, several avenues are open. Good chip thickness screening is essential, so that only chips of suitable thickness (usually less than 8 mm) reach a digester. The overthick stream in a chip thickness screening plant goes to an air density separator in which particles of higher density than the too-thick wood chips fall out. Fortunately, most of the biological knots are purged here and go elsewhere, usually to a hog fuel boiler or a landfill. The overthick chips are sliced or crushed to suitable effective thickness and join the accepts from chip screening.

Inevitably, though, some overthick chips and biological knots find their way to kraft digesters, and subsequently contribute to the rejects streams from knotters. The problem is amplified if there is no chip thickness screening plant, or when it is not operating efficiently. The research described here addresses the fate of these hard-to-pulp particles in order to make rational decisions about what to do with them in kraft pulp mills. We studied two cases: Case 1, beginning with stockpiled knotter rejects from a mill, and Case 2, starting with biological knots captured by an air density separator in another mill. We employed conventional pilot-plant kraft cooking to determine the kraft pulpability of these materials.

EXPERIMENTAL METHODS

Case 1 – We hand-sorted stockpiled softwood knotter rejects from Mill 1 into three main categories: hard, round-edged "Biological Knots" of any size, "Pulpable?" wood chips of any size (this material had already experienced some cooking, but we had no idea about its real pulpability), and rocks. Significant amounts of semi-fibrous, pulp-like material still clung to many of the solid wood particles. All of the material was very wet because it had been stockpiled outdoors.

Separate 200 g (oven-dry basis) samples of the Pulpable? and Biological Knots materials were pulped in 2L bombs using normal kraft cooking conditions from Mill 1: 18.5% effective alkali (as Na₂O on wood); 34.9% sulphidity (on an active alkali basis); 4:1 liquor:wood (L/kg); 90 minutes to 171°C maximum temperature; and a range of H-factors to reach kappa numbers between 10 and 40. After pulping, the cooked material was disintegrated at low consistency in diluted black liquor under mild agitation for two minutes, then screened on a vibrating flat screen having 0.25 mm wide slots.

Case 2 – Rejects from the air density separator in Mill 2 were hand-sorted into three categories: biological knots of any size; overthick wood chips of any size, and rocks. The overthick wood chips and rocks were discarded. The mill was operating on Douglas fir chips.

To determine the fate of biological knots when pulping them more than once, two separate 1.0 kg charges (oven-dry basis) were used in each cook in a 20L digester having forced liquor circulation. The kraft cooking conditions from Mill 2 were as follows: 16.0% effective alkali (as Na₂O on wood); 35.0% sulphidity (on an active alkali basis); 4:1 liquor:wood (L/kg); 90 minutes to 170°C maximum temperature; and an H-factor of 1745 to reach a kappa number of 38 in the screened pulp from the first generation of pulping. After pulping, each batch of cooked material was washed twice with 6L aliquots of tap water at 80°C, then "screened" through a static cylindrical basket having 10 mm diameter holes while agitating the solid material with a high-pressure stream of water (thereby simulating the separation occurring in a pressure knotter). The material not passing through the holes was designated knotter rejects, and was used in the next generation of pulping. The material which passed through the holes was screened on a vibrating flat screen having 0.25 mm wide slots; the fraction passing through the screen was designated the screened pulp yield, and that retained on the screen was the screen rejects.

The same procedure was repeated in the Generation 2 and 3 cooks, starting each time with the

previous generation's knotter rejects. Ten 1.0 kg samples of fresh biological knots were cooked in Generation 1, three 1.0 kg samples of first-generation knotter rejects were cooked in Generation 2, and one 1.0 kg sample of second-generation knotter rejects was cooked in Generation 3.

Screened pulps from Generations 1, 2, and 3 were tested for physical properties according to PAPTAC Standard Methods.

RESULTS AND DISCUSSION

Case 1 – Figure 2 shows the procedure for hand-sorting the stockpiled knotter rejects. On a dry basis, about one-third of the material was Biological Knots (~30% solids content), almost two-thirds was Pulpable? (~20% solids), and 2-3% was rocks. Having been stockpiled outdoors in a damp climate, all of this material was considerably wetter than the normal wood chips at Mill 1. There were at least two reasons for this: (i) the knotter rejects had already been exposed to one cycle of kraft impregnation and cooking, and (ii) the fibrous material accompanying them and clinging to their surfaces would naturally absorb and retain moisture from precipitation.

To obtain more information on the semi-fibrous material clinging to the surfaces of the Pulpable? particles, we treated a sample for one minute at ~2% consistency in a British disintegrator, then screened the product on a vibrating flat screen having 0.25 mm wide slots. Screen rejects accounted for 55% of the sample; the remaining 45% was pulp with a kappa number of 41.

Pulping rate and yield were determined; pilot-plant cooking of normal chips from Mill 1 served as the baseline. Figure 3 shows the pulping rates. The Pulpable? fraction delignified much faster than normal chips, while the Biological Knots cooked much slower. Normalized to 30 kappa number, the Pulpable? material required an H-factor of 500, normal chips 1200, and Biological Knots 2400. Projected back to 0 H-factor, the Pulpable? curve predicts a kappa number of ~40, indicating that the fraction was predominantly easily-cooked pulp and thin, pre-impregnated chips. Coincidentally, this projected value agreed with the 41 kappa number of the disintegrated, screened Pulpable? material.

Because the rates of delignification in Figure 3 are so different, it is clear that putting knotter rejects into a digester along with normal wood chips will make the pulping far more heterogeneous than cooking the wood chips alone.

The Pulpable? fraction gave significantly higher screened pulp yields than were obtained from normal chips (Figure 4). Because the dominant wood species was Douglas fir, the data were artificially high [7] –

one would expect a screened yield of $\sim 46 \pm 1\%$ at 30 kappa, just as shown by the normal chips. Again, this indicates that the Pulpable? fraction carried a significant amount of easily-delignified, pulp-like material into cooking, thereby raising the yield levels above those expected from pulping wood chips.

With Biological Knots, the screened yields appeared to be similar to those from normal chips: $\sim 45\%$ at 30 kappa. This result was misleading, however – once again, easier-to-cook, semi-fibrous material accompanying these knots into pulping had increased the yields to unrealistic high values.

When rejects were plotted against kappa numbers (Figure 5), the highest values came from Biological Knots and rose exponentially with kappa number, both predictable outcomes. Earlier, Axegård had noted that the pulping of biological knots led to high rejects levels because the knots had higher lignin content and were typically thicker than normal chips [4]. Rejects from pulping Mill 1's normal chips were very low, partly the result of the low kappa number range and partly due to good chip thickness control.

Although rejects from the Pulpable? fraction were intermediate in value, this turned out to be wrong – 80% of these “rejects” were sand particles which we had not been able to see and hence remove in our original hand sorting. In fact, the *wood-based* rejects from the Pulpable? fraction were only 1.6% from wood at kappa 30. Accounting for the (unseen) sand in the Pulpable? starting material which was pulped, the screened yield at 30 kappa rose from 50% to 53%, and the wood-based rejects rose from 1.5% to 1.6%. With this recalculation and the data in Figure 4, we could determine that there was a 6-7% gap (at 30 kappa) between the screened yield from the Pulpable? fraction and that from normal chips. If we applied the same difference to the screened yield from Biological Knots, the value would decrease to 39-40%.

In summary, kraft pulping of biological knots from Mill 1 was much slower than that of normal chips, and resulted in lower screened yields and higher rejects. And because the Biological Knots and Pulpable? fractions carried considerable amounts of easily-cooked, pulp-like material with them into pulping, both the rate and yield results were displaced to higher values than would otherwise be expected.

Case 2 – This time, we began with a drum of rejects acquired from the bunker under Mill 2's air density separator when their chip thickness screening plant was in the middle of a run on Douglas fir chips. Hand sorting showed that the air density separator rejects contained 38% biological knots of various sizes and averaging 60% solids content, 61% overthick wood

chips, and 1% rocks. Only the biological wood knots were used in Generation 1 of our kraft pulping experiments. The strategy is outlined in Figure 6. The pulping conditions were based on those used to make bleachable-grade Douglas fir pulp in the mill. Ten 1.0 kg samples of fresh biological knots were cooked in Generation 1, three 1.0 kg samples of first-generation knoter rejects in Generation 2, and one 1.0 kg sample of second-generation knoter rejects in Generation 3 (all under identical pulping conditions).

Figure 7 and Table I show the results from all three generations of pulping. The kappa number of the screened pulp rose with each generation, from 38 to 48 to 80, indicating ever more difficult delignification of the recycled knoter rejects. The screened yields from pulping knoter rejects in Generations 2 and 3 were 31-32%, a clear sign that while pulp was produced in each generation, the yield values were more than 15% lower than those from normal Douglas fir chips. The total yields (screened yield + screen rejects + knoter rejects) decreased from 56% to 51% to 50% across the three generations.

In Generations 2 and 3 in which knoter rejects were pulped, next-generation knoter rejects were produced in comparable yields: 19% in Generation 2 and 16% in Generation 3. This confirms that the pulping of previously cooked and recycled biological knots is not a good practice – the material continues to resist delignification across several generations, resulting in poor pulp yields and large amounts of rejects.

If we now assume 100% *knotter rejects* to start and an average yield of 17.5% of knoter rejects per cook, we can calculate the following sequence:

- Generation I 17.5% yield of knoter rejects
- Generation II 3.1% yield of knoter rejects (i.e., from Gen.I knoter rejects)
- Generation III 0.5% yield of knoter rejects (i.e., from Gen.II knoter rejects)
- Generation IV 0.1% yield of knoter rejects (i.e., from Gen.III knoter rejects)
- Generation V trace of knoter rejects

Thus, to reduce knoter rejects to a negligible amount requires at least four cooks, and the half-life of these Douglas fir knoter rejects is at least two complete kraft cooks.

If, instead, we calculated such a progression beginning with *biological wood knots* (having a first-generation knoter rejects yield of 31%, Figure 7), then five cooks would be needed to get below a yield of 0.1% knoter rejects.

These results clearly demonstrate that the statement “Biological knots primarily become

dissolved solids in the black liquor rather than pulp” [1] is not correct, any more than is the once-popular misconception that kraft pulping of sawdust produces dissolved organics but no pulp.

In Case 2, we also examined the physical properties of the screened pulps from the three generations of cooking. Length-weighted average fibre length was 2.83 mm in the pulp from normal Mill 2 chips. In the pulps from biological knots and their knoter rejects descendants, average fibre length was much lower: 1.51 mm in Generation 1, 1.16 mm in Generation 2, and 1.07 mm in Generation 3 (Figure 8).

Bauer-McNett fractionation showed similar results (Figure 9). Whereas 63% of the pulp from normal chips was in the R14 fraction, the pulps from biological knots had R14 values of 14% (Generation 1), 3% (Generation 2), and 2% (Generation 3), accompanied by much larger amounts of shorter-fibred 14/28, 28/48, and 48/100 material. After Generation 3, the Bauer-McNett distribution more closely resembled that of a hardwood pulp than a softwood one.

Not surprisingly for ever more difficult pulping, unbleached pulp brightness also decreased across the generations of cooking: 27% ISO in pulp from normal chips versus 26% (Generation 1), 21% (Generation 2), and 16% (Generation 3).

Tear-tensile analysis of the pulps from biological knots and re-cooked knoter rejects demonstrated that substantially weaker pulps came from this type of wood material, and that they got still worse across the generations (Figure 10). Even the Generation 1 pulp had only half the tearing resistance (at mid-range breaking length) of pilot-plant pulps from normal wood chips. Freeness-tensile analysis (Figure 11) showed a similar picture – there was a large gap between pulp from normal chips and that from cooking fresh biological knots, and further decreases occurred across the generations of re-cooking of knoter rejects. Axegård also found that re-cooking of screen rejects (including biological knots) led to inferior pulp strength [4].

Together, these comparisons of the physical properties of the unbleached pulps from biological knots showed that they were significantly weaker than pulp from normal chips, and that they became progressively worse in each generation of re-cooking. In a kraft mill, therefore, the recycling and re-cooking of knoter rejects derived from biological knots along with normal chips has the potential to make overall delignification much more heterogeneous, reduce the pulp yield, and severely impair average fibre length and pulp strength.

In fact, there is nothing good to say about the practice of re-cooking knotted rejects derived from biological knots. Instead, biological knots should be purged as efficiently as possible from chip furnishes going to kraft digesters, and knotted rejects containing cooked biological knots should be kept away from digesters.

CONCLUSIONS

Compared with normal wood chips, biological knots cook poorly in conventional kraft conditions, and produce large amounts of knotted rejects (30% in these experiments on Douglas fir). When the knotted rejects generated from the pulping of biological knots are repeatedly re-cooked, they continue to generate significant amounts of knotted rejects (17.5% on average, per generation).

The half-life of Douglas fir biological knots is at least two complete kraft cooks; to eliminate knotted rejects requires at least four cooks.

Across the generations of re-cooking knotted rejects derived from biological knots, the screened pulps show a clear progression in characteristics: increasing kappa number, and large decreases in average fibre length, Bauer-McNett R14 fraction, unbleached brightness, tear-tensile strength, and freeness.

These results demonstrate that the re-cooking of biological knots and their knotted rejects descendants together with normal chips makes kraft pulping far more heterogeneous and penalizes pulp yield and pulp quality. It is a practice to be avoided.

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TABLE I. KRAFT PULPING RESULTS FROM COOKING FRESH BIOLOGICAL KNOTS (GENERATION 1) AND THEN RE-COOKING THE KNOTTER REJECTS DERIVED FROM THEM ^a (GENERATIONS 2 AND 3).

	<u>Generation 1</u>					Avg of 10	<u>Generation 2</u>		Avg of 3	<u>Gen. 3</u>	
Kappa number											
Top	34.1	39.0	43.6	36.5	40.0		47.1				
Bottom	36.6	40.8	34.9	39.4	36.1	38.1	51.7	44.4	47.7	79.8	
Knotter rejects ^b , %											
Top	30.3	32.4	29.5	30.7	29.3		21.2				
Bottom	30.4	33.5	30.9	29.1	28.7	30.5	16.3	19.7	19.1	15.8	
Screen rejects, %											
Top	0.45	0.47	0.79	0.61	0.65		0.72				
Bottom	0.55	0.55	0.39	0.88	0.61	0.60	1.38	0.83	0.98	2.26	
Screened yield, %											
Top	24.5	23.3	26.9	24.7	25.1		30.0				
Bottom	25.3	23.8	24.1	25.0	25.2	24.8	32.1	30.5	30.9	31.7	

^a Cooks were done in a 20L digester with forced liquor circulation; In each cook, two 1.0 kg wood charges were placed in perforated baskets one on top of the other. Standard pulping conditions: 16.0% effective alkali (as Na₂O on wood); 35.0% sulphidity (on an active alkali basis); 4:1 liquor:wood (L/kg); 90 minutes to 170°C maximum temperature; H-factor 1745.

^b Knotter rejects = cooked material not passing through 1 cm diameter round holes. The values in italics were calculated from the wet weights actually measured; the other values were determined by drying to constant weight.



Figure 1. Biological wood knots are derived from chipping a part of a tree stem into which a branch was sticking. Their cores tend to be round in cross-section, higher in density than normal stem wood from the same tree, darker in colour, and more heavily lignified. Knots are surrounded by reaction wood, which is also different from stem wood. (The twoonie is 28 mm in diameter).

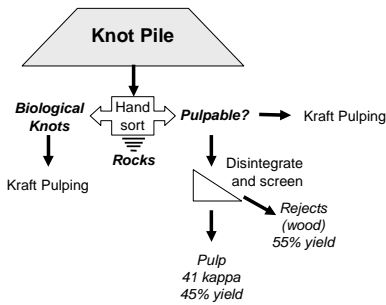


Figure 2. Stockpiled knottor rejects were hand-sorted into three fractions: Biological Knots, Pulpable? material (which, by observation alone, had uncertain pulpability), and rocks. In one case, we disintegrated and screened a Pulpable? sample to determine how much of it was already pulp-like.

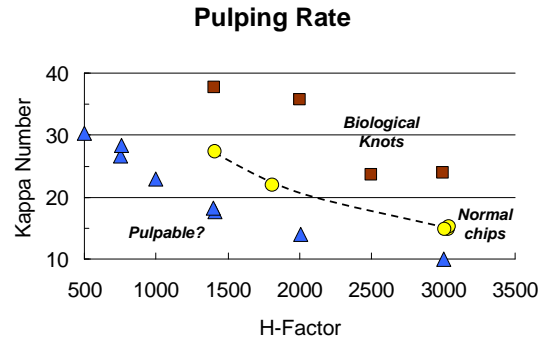


Figure 3. Relative to normal Douglas fir chips from the same mill, the Pulpable? material delignified much faster and the Biological Knots much slower.

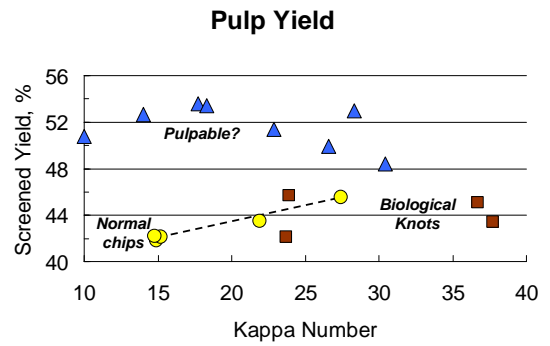


Figure 4. Relative to normal Douglas fir chips from the same mill, the Pulpable? fraction provided much high pulp yield at a given kappa number, but the values were artificially inflated by the pulp-like material accompanying it into pulping. So were the yields from the Biological Knots.

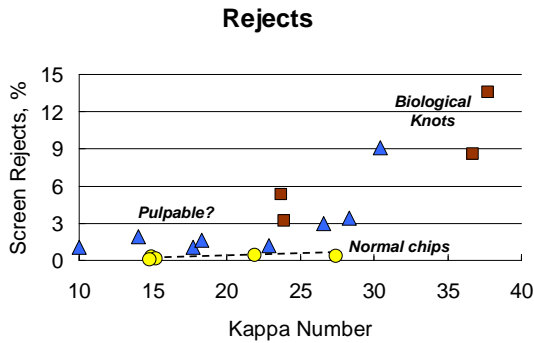


Figure 5. Pulping of Biological Knots generated high amounts of screen rejects, and the values increased exponentially with kappa number. Although the Pulpable? fraction appeared to have a serious rejects problem too, in this case 80% of the rejects turned out to be sand.

Kraft Pulping of Biological Knots and Knotter Rejects – Results

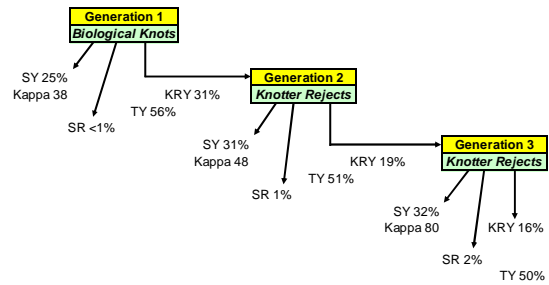


Figure 7. The experimental results showed that the fresh biological knots cooked poorly, giving an average screened pulp yield of 25% and an average knotter rejects yield of 31%. In Generation 2, re-cooking of the knotter rejects from Generation 1 provided a screened yield of 31% and a knotter rejects yield of 19%. In Generation 3, re-cooking of the knotter rejects from Generation 2 provided a screened yield of 32% and a knotter rejects yield of 16%. By calculation, the half-life of these knotter rejects was two complete cooks.

Kraft Pulping of Biological Knots and Knotter Rejects – Strategy

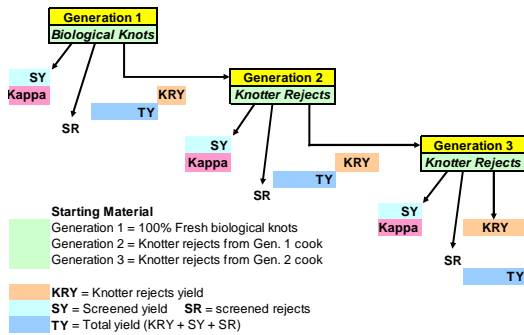


Figure 6. Our strategy for examining the kraft pulpability of biological knots is outline here. Ten Generation 1 cooks of fresh biological knots from Mill 2 provided the “knotter rejects” for three Generation 2 cooks, which in turn provided the knotter rejects for one Generation 3 cook. This way, we could determine what happens in the re-cooking of knotter rejects derived exclusively from biological wood knots.

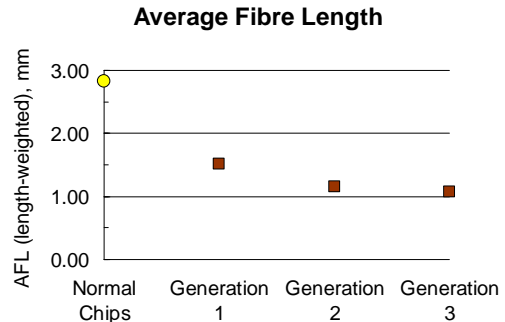


Figure 8. Average fibre length in the screened pulps made from biological knots was far lower than in pulp from normal Douglas fir chips, and it declined in each generation of cooking.

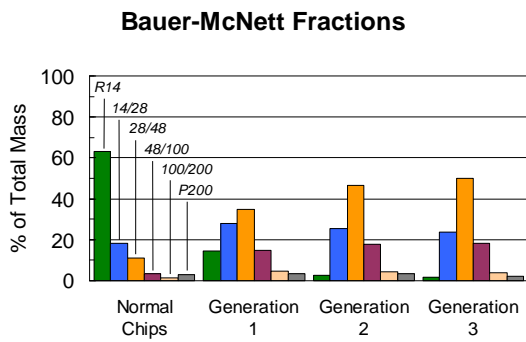


Figure 9. Bauer-McNett fractionation showed that the screened pulps made from biological knots had their entire distributions shifted significantly to the right. Whereas pulp from normal chips had an R14 fraction of 63%, the R14 content of Generation 1 pulp from biological wood knots was only 14%, and then dropped below 3% in the re-cooking of the knotted rejects.

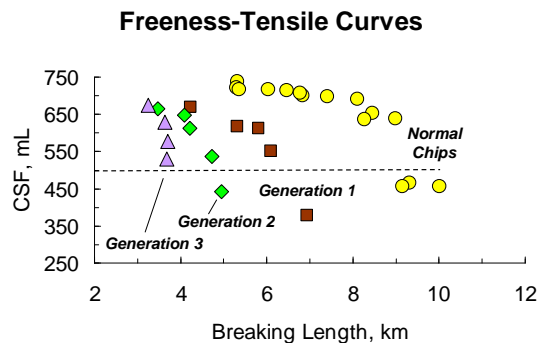


Figure 11. In freeness versus tensile strength, there were four distinct cases in declining order: pulp from normal chips, followed by the pulps from the successive generations cooking of biological knots.

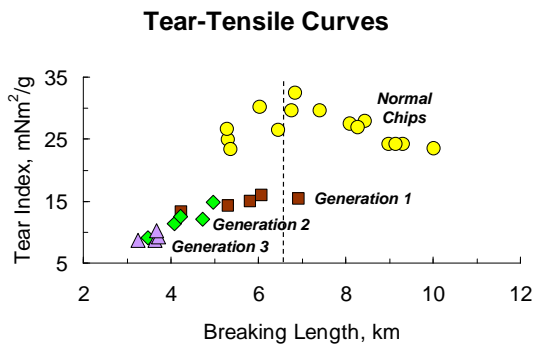


Figure 10. In tear-tensile strength, the pulps made from biological knots were far weaker than those from normal chips. Overall strength declined further across the generations of re-cooking.