

A functional environmentally safe plastic, designed for the circular economy

Aquapak's Hydropol™ is a specially engineered material that can create a range of packaging products that are designed to eliminate plastic pollution at source

Repulping

Aquapak has successfully developed intermediate hydrolysis grades which demonstrate excellent adhesion to paper and paperboard with enhanced barrier properties. The Hydropol 33 series grades have been independently tested for repulpability (mimicking a standard paper recycling mill typically at a repulping temperature of 40°C) and have all passed the testing protocols.

Summary of findings:

- Hydropol 33100 (33100 represents the same chemistry as the 33 series) coated paper disintegrates with complete dispersion of fibres when repulped at 40°C and above (coating is almost non-detectable when repulped at 20°C) -meets criteria outlined in ISO 5263-1 Laboratory Disintegration of chemical pulps
- No evidence of Hydropol coating on mesh 8 with Bauer McNett fractionation-indicates complete polymer dissolution and fibre dispersion
- Solubilised Hydropol at high loading rates appears to have no effect on paper sheet properties in the case of recycled grey board furnish. For virgin copier paper furnish, no effects were noted for bulk, roughness (bottom), tear index, drainage and Schopper. Slight changes were noted for porosity, roughness (top), burst index, tensile index and freeness
- Hydropol coated paper would repulp and release valuable fibre for paper and board manufacturing
- Hydropol coating would dissolve and would NOT form part of 'mill rejects stream' (Note: valuable fibre is entrained and lost with mill rejects (yield loss); rejects are costly to dispose for mills without waste to energy systems)
- Hydropol coating appears not to be substantive to fibre and unlikely to contribute either positively or negatively to paper sheet properties



- Hydropol coated paper could be recycled with Mixed Paper or OCC at packaging mills
- Segregated Hydropol coated paper e.g. coated white food board could be recycled at virgin-fibre based mills
- Effluent from Grey board+ Hydropol coated paper (20%) did not inhibit activated sludge bacterial respiration rates (i.e. no acute toxicity)
- Treatability of Hydropol by mill effluent treatment systems (DAF, AD and activated sludge)
- Laboratory tests showed that PVOH is slowly biodegraded under anaerobic conditions (34.3% reduction over 28 days), and is almost completely removed under aerobic conditions (99.3% reduction over 28 days)

Please see the appendix below for the full report.

Aquapak is working in several projects with global brands, major converters and waste and recyclers to demonstrate the above recycling and recovery solutions at scale. Whilst many of these projects are commercially sensitive Aquapak will provide information and results where possible.

Dr Elizabeth Smith

Technical Manager



Appendix







Repulpability assessments of Hydropol coated papers

Updated September 2020



Objectives

- 1. To confirm repulpability of Hydropol coated papers
- 2. To determine the impact of solubilised Hydropol coating on paper sheet properties (strength etc)
- 3. Evaluation of deposition and sticky forming potential of Hydropol extrusion coated papers
- 4. Fate of Hydropol within papermill wastewater treatment systems



Approach

Repulpability - aimed at providing data to confirm rate of fibre dispersion, influence of dispersion parameters such as temperature, pH and fractionation to determine fibre yield and quantify non-repulpable components (rejects)

Recyclability – quantification of the useable fibre fraction and its 'paper making potential'

Note: No EU standard is available to assess universally the repulpability and recyclability of paper products. The protocol developed here combines various pulp and paper standards and knowledge of mill repulping and fibre clean up stages to assess the repulpablity and recyclability of Hydropol-coated papers



Test samples





Control 'base paper' (L) – chemical pulp, 72gsm coated on one side with modified starch. Hydropol 33100 (R)- 30μ m coating applied on 'base paper' – sample cut to $10cm \times 5cm$ for repulping tests



1) Repulpability - ISO 5263-1 Laboratory Disintegration of chemical pulps



- Temperature 20°C, 40°C and 50°C (temperatures typical of 'open' and 'closed' pulper systems)
- pH 7.1
- Disintegrator revolutions 10,000 and 50,000
- Hydropol coated paper tested as 100% of fibre furnish
- Fibre dispersion Y/N
- · pH measured after pulping



Laboratory scale repulpability assessments







Laboratory disintegrator, temperature checks and charging pulper with test sample



Repulpability – fibre fractionation (Tappi T233 cm 06)

- Samples fractionated using a Bauer McNett fractionator (Tappi T233 cm-06) equipped with 8/16/50 and 100 mesh screens (slot size-2.5mm/1.5mm/0.31mm and 0.15mm)
 - Weight of fraction retained by each mesh measured
 - Examination for presence of Hydropol coating retained by each mesh
- Undispersed fibre/coating is retained by mesh 8, while softwood and hard wood fibres are retained by mesh 50 and 100 respectively
- Material passing through 100 mesh would be lost to drain/effluent





Results – impact of temperature and repulping duration

Temperature	Disintegrator Revs.	pH (start)	pH (end)	Fractionation				
				Percent Retention				
°C	K			8	16	50	100	>100
20	10	7.1	8.89	2.82	18.64	1.97	32.02	44.55
20	10	7.1	8.84	2.21	6.82	1.28	9.11	68.72
20	50	7.1	8.86	0.55	0.14	1.13	10.03	88.15
40	10	7.1	8.11	0.90	4.90	1.30	14.10	78.80
40	50	7.1	8.45	0.20	5.30	1.20	12.30	81.00
50	10	7.1	8.92	0.74	8.06	1.55	17.22	72.43
50	50	7.1	8.91	0.50	10.06	1.25	21.16	67.24
	°C 20 20 20 40 40 50	20 10 20 10 20 50 40 10 40 50 50 10	°C K 20 10 7.1 20 10 7.1 20 50 7.1 40 10 7.1 40 50 7.1 50 10 7.1	°C K 20 10 7.1 8.89 20 10 7.1 8.84 20 50 7.1 8.86 40 10 7.1 8.11 40 50 7.1 8.45 50 10 7.1 8.92	°C K 8 20 10 7.1 8.89 2.82 20 10 7.1 8.84 2.21 20 50 7.1 8.86 0.55 40 10 7.1 8.11 0.90 40 50 7.1 8.45 0.20 50 10 7.1 8.92 0.74	°C K 8 16 20 10 7.1 8.89 2.82 18.64 20 10 7.1 8.84 2.21 6.82 20 50 7.1 8.86 0.55 0.14 40 10 7.1 8.11 0.90 4.90 40 50 7.1 8.45 0.20 5.30 50 10 7.1 8.92 0.74 8.06	°C K 8 16 50 20 10 7.1 8.89 2.82 18.64 1.97 20 10 7.1 8.84 2.21 6.82 1.28 20 50 7.1 8.86 0.55 0.14 1.13 40 10 7.1 8.11 0.90 4.90 1.30 40 50 7.1 8.45 0.20 5.30 1.20 50 10 7.1 8.92 0.74 8.06 1.55	°C K 8 16 50 100 20 10 7.1 8.89 2.82 18.64 1.97 32.02 20 10 7.1 8.84 2.21 6.82 1.28 9.11 20 50 7.1 8.86 0.55 0.14 1.13 10.03 40 10 7.1 8.11 0.90 4.90 1.30 14.10 40 50 7.1 8.45 0.20 5.30 1.20 12.30 50 10 7.1 8.92 0.74 8.06 1.55 17.22

- Uncoated control after 10,000 revolutions, 2.82% of control material was retained by mesh 8 indicating good fibre dispersion. 32% retained by mesh 100 suggesting high hardwood fibre content. pH increased to 8.89
- Hydropol coated samples at 20°C and 50,000 revolutions only a small amount of Hydropol was detected on mesh 8 with complete fibre dispersion. At 40°C and 50°C and 10,000 or 50,000 revolutions, NO Hydropol was noted on mesh 8 with complete fibre dispersion.
- pH increases noted to be similar to uncoated control

<u>Summary</u>: Hydropol coating is repulpable with complete fibre dispersion at <u>40°C</u> and above with complete solubilization of the coating



2) Impacts of Hydropol coating on paper sheet quality parameters

- 1. Could Hydropol-coated packaging papers be used in CCM and other packaging grades
- 2. Does Hydropol impact on recycled paper sheet properties?
- 3. Could Hydropol serve as a 'strength enhancer' enabling mills to use lower quality recovered fibre?
- 4. Could Hydropol-coated papers be used in fine papers (if segregated) so that products can qualify 'recycled fibre content'?

1 Grey board	1 Virgin fibre
2 Greyboard + uncoated control (20%)*	2 Virgin fibre + uncoated control (20%)
3 Greyboard + uncoated control (50%)	3 Virgin fibre + uncoated control (50%)
4 Greyboard + Hydropol coated paper (20%)*	4 Virgin fibre + Hydropol coated paper (20%)
5 Greyboard + Hydropol coated paper (50%)	5 Virgin fibre + Hydropol coated paper (50%)

*Samples of effluent tested by AquaEnviro for effluent treatability



Impacts of Hydropol-coated paper on greyboard

	Fibre type		Furnish					
	Greyboard		100	80	80	50	50	
	Uncoated			20		50		
	Hydropol coated				20		50	
		Units						Change-compared with inclusion
Test								of uncoated control
Bulk		cm³/g	1.966	1.908	1.911	1.810	1.881	No change
Porosity		mls/min	1109	1308	1277	1788	1792	No change
Roughness	Glazed top	μm	4.49	4.52	4.59	4.93	4.96	No change
	Bottom	μm	6.95	7.03	6.96	7.44	7.10	No change
Burst Index		Kpa/g	1.18	1.20	1.19	1.41	1.35	No change
Tear Index		mNm²/g	4.83	4.93	4.70	5.17	5.17	No change
Tensile Index		Nm/g	25.59	27.89	28.06	29.94	28.15	No change
Drainage		secs	8.29	8.06	8.36	6.17	6.95	No change
Freeness		Csf	337	318	340	337	332	No change
Schopper		°SR	36	38	36	37	37	No change

Inclusion of either the uncoated control or Hydropol coated papers @20 or 50% w/w <u>imparted the same</u> changes to the paper sheet properties.



Impacts of Hydropol-coated paper on virgin copier paper

	Fibre type			Furnish				
	Virgin copier paper		100	80	80	50	50	
	Uncoated			20		50		
	Hydropol coated				20		50	
Test		Units						Change-compared with inclusion of uncoated control
Bulk		cm³/g	1.888	1.815	1.872	1.829	1.773	No change
Porosity		mls/min	3405	3487	4376	3897	4372	Slight increase in porosity
Roughness	Glazed top	,	5.03	5.08	5.30	5.07	5.27	Slight increase in roughness
	Bottom	μm	7.06	7.03	7.04	7.01	7.07	No change
Burst Index		Kpa/g	1.34	1.65	1.35	1.51	1.38	Slight strength decrease
Tear Index		mNm²/g	4.53	5.57	4.70	5.07	5.03	No change
Tensile Index		Nm/g	30.08	32.42	29.69	32.11	29.51	Slight reduction in tensile index
Drainage		secs	5.26	4.87	5.09	5.04	5.04	No change
Freeness		Csf	392	407	428	392	443	Increased freeness
Schopper		°SR	32	31	30	32	29	No change

Inclusion of uncoated control or Hydropol coated papers @20 or 50% w/w imparted the same changes to bulk, roughness (bottom), tear index, drainage and Schopper. Slight changes were noted for porosity, roughness (top), burst index, tensile index, and freeness.



Impacts on paper sheet quality parameters

- Could Hydropol-coated packaging papers be recycled into CCM and other packaging grades? YES. Uncoated control and Hydropol coated papers imparted the same paper sheet properties to greyboard
- 2. Does solubilized Hydropol impact on recycled paper sheet properties? NO. Uncoated control and Hydropol coated papers imparted the same paper sheet properties
- 3. Could solubilized Hydropol impart strength to fibre/sheet properties enabling mills to use lower quality recovered fibre? **NO**
- 4. Could Hydropol-coated papers be used in fine papers (if segregated) so that products can qualify 'recycled fibre content'? **YES**

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3) Evaluation of deposition and sticky forming potential of Hydropol extrusion coated papers

BS ISO 15360 Recycled pulps – Estimation of stickies and plastics – Part 1: Visual method

Principle: disintegrated pulp sample is passed through a laboratory screen (slit size- $100\mu m$). The material retained on the screen is transferred to a filter paper, heated under pressure and sticky* particles identified and their number and area estimated.

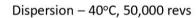
Test material – Testliner (85gsm) with Hydropol extrusion coating (15μm)

*Stickies – diverse group of materials that are retained on a laboratory screen of given slit aperture (100 or $150\mu m$) and which adhere to objects which they touch. Stickies may adhere to objects at ambient temperature or they may adopt adhesive characteristics when subjected to elevated temperatures, elevated pressure or change in pH







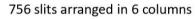


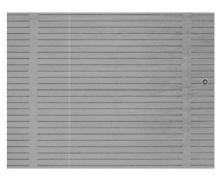


Somerville screen









Slit width- $100\mu m$



Results: Hydropol-coated test liner

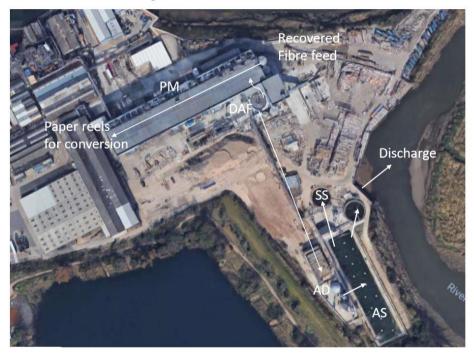




Filter papers heated between two stainless steel discs at 150°C for 5 mins with 1kg weight on discs. Some fibre bundles noted. No stickies detected



4) Fate of Hydropol within papermill wastewater treatment systems



Generic example:

Test liner manufacturer (Furnish – OCC and selected Mixed Papers)

1 Primary treatment – Dissolved Air Flotation – recovers fibre, cellulose fines and ash back to the mill

2 Secondary biological treatment -mineralises dissolved organic load:

i. anaerobic digestion (AD)

ii. aerobic activated sludge (AS)

iii. sludge settlement (SS)

iv. Treated effluent discharged to river



Fate of Hydropol within papermill wastewater treatment systems

Biomethane potential test - Determines treatability by anaerobic digestion and conversion into valuable biogas

• **34.3**% of PVOH was biodegraded to biogas under anaerobic conditions over the 28 days test period

Inherent aerobic biodegradability OECD 301B BCOD Test (Zahn Wellens)
Quantifies the biodegradability of soluble organic material within an effluent

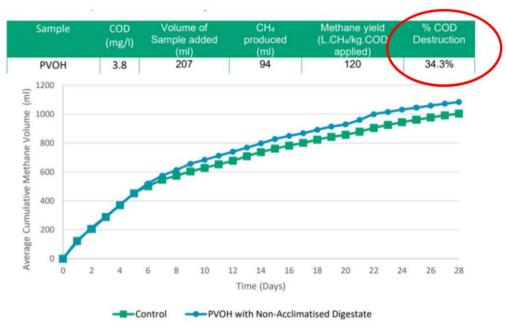
• PVOH was almost completely removed in non-acclimatized tests with **99.3%** COD reduction over the 28 days test period

Carbonaceous and nitrification inhibition test provides a measure of the treatability of the effluent samples and if is in inhibitory to aerobic activated sludge bacterial respiration

• No inhibition of bacterial respiration rate (ie absence of acute toxicity)



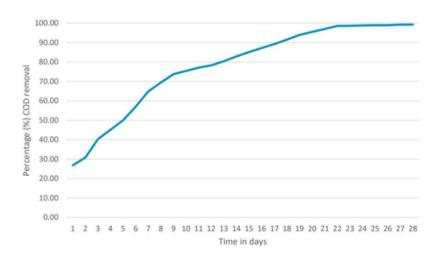
Feedstock parameters and methane evolution profile



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Percentage soluble COD (PVOH) removal for the non – acclimatized aerobic mixed liquor tests



99.3% COD removal



Fate of Hydropol within papermill wastewater treatment systems

Conclusions

- Laboratory tests provide strong evidence that PVOH is slowly biodegraded under anaerobic conditions but is almost completely removed under aerobic conditions
- Pilot testing would be required to determine whether biodegradability would be replicated under the process conditions of the full-scale effluent plant and whether Hydropol could be treated without any other impacts upon the process



Summary

Repulpable

- Hydropol 33100 coated paper disintegrates with complete dispersion of fibres when repulped at 40°C and above (coating is almost non-detectable when repulped at 20°C) - meets criteria outlined in ISO 5263-1 Laboratory Disintegration of chemical pulps
- No evidence of Hydropol coating on mesh 8 with Bauer McNett fractionation-indicates complete polymer dissolution and fibre dispersion for papermaking

Recyclable

Complete fibre dispersion for papermaking. Solubilised Hydropol at high loading rates
appears to have no effect on paper sheet properties in the case of recycled grey board
furnish. For virgin copier paper furnish, no effects were noted for bulk, roughness (bottom),
tear index, drainage and Schopper. Slight changes were noted for porosity, roughness (top),
burst index, tensile index and freeness



Summary (continued)

- Potential for deposition
 - Sticky tests demonstrate low potential for deposition within mill processes following solubilization during the repulping process
- Biodegradability
 - Laboratory tests showed that PVOH is slowly biodegraded under anaerobic conditions but is almost completely removed under aerobic conditions.



At a mill

- 1. Hydropol coated paper would repulp and release valuable fibre for paper and board manufacturing
- 2. Hydropol coating would dissolve and would NOT contribute to the 'mill rejects stream'. Note: valuable fibre is entrained and lost with mill rejects (yield loss); rejects are costly to dispose for mills without waste to energy
- 3. Hydropol coating appears not to be substantive to fibre and unlikely to contribute either positively or negatively to paper sheet properties
- 4. Hydropol coated paper could be recycled with Mixed Paper or OCC at packaging mills



At a mill (continued)

- 5. Segregated Hydropol coated paper eg coated white food board could be recycled at virgin-fibre based mills
- 6. Hydropol does not generate stickies following the repulping process
- 7. Effluent from Greyboard + Hydropol coated paper (20%) did not inhibit activated sludge bacterial respiration rates (ie no acute toxicity)
- 8. Hydropol entering the mill effluent would likely be partially treated by AD and almost completely removed following aerobic activated sludge treatment

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