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(54) Hydrophilic dispersion polymers for paper applications

Hydrophile Dispersionspolymere für Papieranwendungen

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(73) Proprietor: **NALCO CHEMICAL COMPANY**
Naperville
Illinois 60563-1198 (US)

(72) Inventors:
 • **Wong Shing, Jane B.**
Aurora,
Illinois 60504 (US)

• **Hurlock, John R.**
Hickory Hills,
Illinois 60457 (US)

(74) Representative: **Wibbelmann, Jobst**
Wuesthoff & Wuesthoff,
Patent- und Rechtsanwälte,
Schweigerstrasse 2
81541 München (DE)

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EP-A- 0 308 752 **EP-A- 0 525 751**
EP-A- 0 821 099 **US-A- 4 696 962**
US-A- 5 334 679

EP 0 831 177 B1

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Description**Background of the Invention**5 **1. Field of the Invention**

[0001] The invention relates to the field of papermaking, and, in particular, to an improved papermaking process utilizing hydrophilic dispersion polymers as retention and drainage aids.

10 **2. Description of the Prior Art**

[0002] In the manufacture of paper an aqueous cellulosic suspension or slurry is formed into a paper sheet. The cellulosic slurry is generally diluted to a consistency (percent dry weight of solids in the slurry) of less than 1 percent, and often below 0.5 percent, ahead of the paper machine, while the finished sheet must have less than 6 weight percent water. Hence, the dewatering aspects of papermaking are extremely important to the efficiency and cost of the manufacture.

[0003] The least costly dewatering method is drainage, and thereafter more expensive methods are used, including vacuum pressing, felt blanket blotting and pressing, evaporation and the like, and any combination of such methods. Since drainage is both the first dewatering method employed and the least expensive, improvement in the efficiency of drainage will decrease the amount of water required to be removed by other methods and improve the overall efficiency of dewatering and reduce the cost thereof.

[0004] Another aspect of papermaking that is extremely important to the efficiency and cost of manufacture is retention of furnish components on and within the fiber mat being formed during papermaking. A papermaking furnish contains particles that range in size from about the 2 to 3 millimeter size of cellulosic fibers to fillers measuring only a few microns. Within this range are cellulosic fines, mineral fillers (employed to increase opacity, brightness and other paper characteristics) and other small particles that generally, without the inclusion of one or more retention aids, would pass through the spaces (pores) between the cellulosic fibers in the fiber mat being formed.

[0005] One method of improving the retention of cellulosic fines, mineral fillers and other furnish components on the fiber mat is the use of a coagulant/flocculant system, added ahead of the paper machine. In such a system there is first added to the furnish a coagulant, for instance a low molecular weight cationic synthetic polymer or a cationic starch which coagulant generally reduces the negative surface charges present on the particles in the furnish, particularly cellulosic fines and mineral fillers, and thereby agglomerates such particles. The coagulant is followed by the addition of a flocculant. The flocculant is generally a high molecular weight cationic or anionic synthetic polymer which bridges the particles and/or agglomerates, from one surface to another, binding the particles into large agglomerates. The presence of such large agglomerates in the furnish increases retention. The agglomerates are filtered out of the water onto the fiber web, where unagglomerated particles otherwise would to a great extent pass.

[0006] While a flocculated agglomerate generally does not interfere with the drainage of the fiber mat to the extent that would occur if the furnish were gelled or contained an amount of gelatinous material, when such flocs are filtered by the fiber web the pores thereof are reduced, thus reducing drainage efficiency. Hence, the retention is increased at the expense of decreasing drainage.

[0007] Another system employed to provide an improved combination of retention and dewatering is described in U.S. Patent Nos. 4,753,710 and 4,913,775, inventors Langley et al., issued respectively June 28, 1988 and April 3, 1990. In brief, such method adds to the aqueous cellulosic papermaking suspension first a high molecular weight linear cationic polymer before shearing the suspension, followed by the addition of bentonite after shearing. The shearing generally is provided by one or more of the cleaning, mixing and pumping stages of the papermaking process, and the shearing breaks down the large flocs formed by the high molecular weight polymer into microflocs, and further agglomeration then ensues with the addition of the bentonite clay particles.

[0008] Another system uses the combination of cationic starch followed by colloidal silica to increase the amount of material retained on the web by charge neutralization and adsorption of smaller agglomerates. This system is described in U.S. Patent No. 4,388,150, inventors Sunden et al, issued June 14, 1983.

[0009] U.S. Patent Nos. 5,098,520 and 5,185,062, issued to Begala, describe methods of improving dewatering in a papermaking process.

[0010] U.S. Patent No. 5,334,679 discloses a papermaking process in which water-soluble cationic polymers produced by suspension polymerization are used as retention aids.

[0011] U.S. Patent No. 4,696,962 relates to the use of specific water-soluble cationic copolymers of (meth)acrylamide as retention and dewatering agents in the paper industry. This document explicitly excludes the use of quaternary ammonium salts and pertains to copolymers produced by emulsion polymerization.

[0012] EP 525 751 discloses the use of specific cationic polymer dispersions as a paper chemical in a papermaking

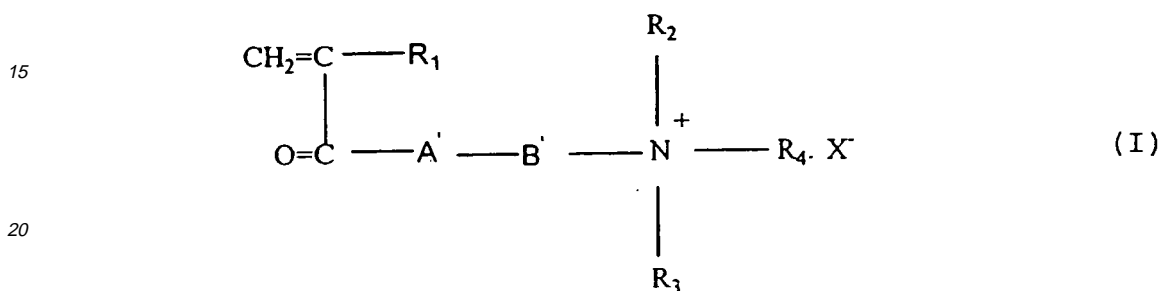
process.

[0013] EP 308 752 relates to a papermaking process using a coagulant, a charged water-soluble acrylamide copolymer as flocculant and colloidal silica.

5 Summary of the Invention

[0014] The present invention relates to the use of a hydrophilic dispersion polymer resulting from the polymerization of monomers comprising:

10 (i) a cationic monomer of the formula (I):



25 wherein R_1 is H or CH_3 ; each of R_2 and R_3 is an alkyl group having 1 to 2 carbon atoms; R_4 is H or an alkyl group of 1 or 2 carbon atoms; A' is an oxygen atom or NH; B' is an alkylene group of 2 to 4 carbon atoms or a hydroxypropylene group; and X^- is an anionic counterion: and

(ii) a second monomer represented by (meth)acrylamide in an aqueous solution of a polyvalent anionic salt to increase retention and/or drainage in a papermaking process comprising:

- 30
- forming an aqueous cellulosic papermaking slurry;
 - adding an effective amount of said hydrophilic dispersion polymer to the slurry;
 - draining the slurry to form a sheet; and
 - drying the sheet
- 35

[0015] The polymerization of the above mentioned monomers may be carried out in the presence of either an organic high-molecular weight multivalent cation comprising a water-soluble polymer containing at least one monomer of formula (I) and/or poly diallyl dimethyl ammonium chloride (DADMAC). After addition of the polymers, the slurry is drained to form a sheet, and the sheet is dried.

40 Brief Description of the Drawings

[0016]

45 FIG. 1 is a graph comparing turbidity reduction between three dispersion polymers and the standard latex retention aid.

FIG. 2 is a graph comparing drainage activity between three dispersion polymers and the standard latex retention aid.

50 FIG. 3 is a graph showing retention activity of higher intrinsic viscosity dispersion copolymers containing 10 and 20 mole % DMAEA-MCQ.

FIG. 4 is a graph showing drainage activity of higher intrinsic viscosity dispersion copolymers containing 10 and 20 mole % DMAEA-MCQ.

55 FIG. 5 is a graph comparing the retention performance of dispersion latex and dry polymers.

FIG. 6 is a graph comparing turbidity reduction of various dispersion polymers with standard flocculants.

FIG. 7 is a graph comparing the drainage activity of various dispersion polymers with standard flocculants.

FIG. 8 is a graph comparing retention performance of dispersion polymers to standard latex polymer.

5 FIG. 9 is a graph comparing drainage performance of dispersion polymers to standard latex polymer.

FIG. 10 is a graph comparing retention performance of dispersion polymers combined with standard coagulants to dispersion polymers alone.

10 FIG. 11 is a graph comparing draining performance of dispersion polymers combined with standard coagulants to dispersion polymers alone.

1 lb = 0.45 kg

15 Description of the Preferred Embodiments

[0017] The invention comprises a papermaking process for improving retention and drainage comprising forming an aqueous cellulosic papermaking slurry and adding a hydrophilic dispersion polymer to the slurry. The slurry is then formed into a sheet and dried.

20 **[0018]** Preferably, the hydrophilic dispersion polymer of the invention is a copolymer of dimethylaminoethyl (meth)acrylate methyl chloride quat (DMAEA-MCQ) cationic monomer and (meth)acrylamide (AcAm). It has been found that the polymer described above confers advantages for use in a papermaking process. Specifically, the hydrophilic dispersion polymers of the invention show improved retention activity compared to dimethylaminoethyl acrylate benzyl chloride quat (DMAEA-BCQ)/acrylamide(AcAm) dispersion copolymer and DMAEA methyl chloride quaternary latex of the same charge. Latex is defined within this application as an inverse water-in-oil emulsion polymer.

25 **[0019]** In an alternative embodiment, the DMAEA-MCQ/AcAm hydrophilic dispersion polymers show nearly equal activity with respect to retention and drainage as compared to the commercial standard latex cationic polymers.

[0020] Examples 1-4 below outline processes for preparing the copolymer at various ratios of the monomer components. Preferably, the amount of dimethylaminoethyl acrylate methyl chloride quaternary present in the copolymer is from about 3 mole percent to about 20 mole percent. Further, the range of intrinsic viscosities for the hydrophilic dispersion polymers of the invention is preferably from about 11.9 to about 21.2 dl/g. According to the preferred method of the invention, the dispersion polymer is added in an amount from about 0.2 to about 2.3 kg of active per 1000 kg of slurry solids (about 0.5 to about 5.0 pounds of active per ton of slurry solids).

30 **[0021]** The present process is believed applicable to all grades and types of paper products and further applicable for use on all types of pulps including chemical pulps, including sulfate and sulfite pulps from both hard and soft woods and acid pulps, thermomechanical pulps, mechanical pulps, recycle pulps and ground wood pulps. Typically, such furnishes will have a pH of from about 3.0 to about 9.0.

35 **[0022]** The following examples are presented to describe preferred embodiments and utilities of the invention and are not meant to limit the invention unless otherwise stated in the claims appended hereto.

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Example 1 - Process for Synthesizing Dispersion Copolymers of Acrylamide and 3 mole % DMAEA-MCQ.

[0023] To a two-liter resin reactor equipped with stirrer, temperature controller and water cooled condenser, was added 287.59 grams of a 48.1 % solution of acrylamide (1.9461 moles), 7.24 grams of an 80.6% solution of DMAEA-MCQ (0.0301 moles), 250 grams of ammonium sulfate, 225.59 grams of deionized water, 27 grams of glycerol, 56.25 grams of a 16% solution of polyDADMAC (poly diallyl dimethyl ammonium chloride)(IV= 1.5 dl/gm), 18 grams of a 20% solution of polyDMAEA-MCQ (IV=2.0dl/gm), and 0.3 grams of EDTA. The mixture was heated to 48°C and 0.50 grams of a 4% solution of 2,2' Azobis(2 amidinopropane) dihydrochloride was added. The resulting solution was sparged with 1000 cc/min. of nitrogen. After 15 minutes, polymerization began and the solution became viscous. Over the next 4 hours the temperature was maintained at 48°C and a solution containing 95.86 grams (0.6487 moles) of 48.1 % acrylamide. 12.07 grams (0.0502 moles) of an 80.6% solution of DMAEA-MCQ, 9 grams of glycerol and 0.1 gram of EDTA was pumped into the reactor using a syringe pump. To the resulting polymer dispersion was added 0.50 grams of a 4% solution of 2,2' Azobis(2 amidinopropane) dihydrochloride. The dispersion was then further reacted for 2.5 hours at a temperature of 48°C to 55°C. The resulting polymer dispersion had a Brookfield viscosity of 5600cps. To the above dispersion was added 10 grams of 99% acetic acid and 20 grams of sodium sulfate. The resulting dispersion had a Brookfield viscosity of 1525 cps and contained 20% of a 97/3 copolymer of acrylamide and DMAEA-MCQ with an intrinsic viscosity of 12.1 dl/gm in 0.125 molar NaNO₃.

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Example 2 - Process for Synthesizing Dispersion Copolymers of Acrylamide and 5 mole % DMAEA-MCQ.

[0024] To a two-liter resin reactor equipped with stirrer, temperature controller, and water cooled condenser, was added 281.68 grams of a 48.1 % solution of acrylamide (1.9061 moles), 12.07 grams of an 80.6% solution of DMAEA-MCQ (0.05023 moles), 250 grams of ammonium sulfate, 225.10 grams of deionized water, 27 grams of glycerol, 33.75 grams of a 16% solution of polyDADMAC (IV=1.5 dl/gm), 36 grams of a 20% solution of polyDMAEA-MCQ (IV=2.0 dl/gm), and 0.3 grams of EDTA. The mixture was heated to 48°C and 0.50 grams of a 4% solution of 2,2' Azobis(2 amidinopropane) dihydrochloride was added. The resulting solution was sparged with 1000 cc/min. of nitrogen. After 15 minutes, polymerization began and the solution became viscous. Over the next 4 hours the temperature was maintained at 48°C and a solution containing 93.89 grams (0.6354 moles) of 48.1 % acrylamide, 20.11 grams (0.08368 moles) of an 80.6% solution of DMAEA-MCQ, 9 grams of glycerol and 0.1 grams of EDTA was pumped into the reactor using a syringe pump. To the resulting polymer dispersion was added 0.50 grams of a 4% solution of 2,2' Azobis(2 amidinopropane) dihydrochloride. The dispersion was then further reacted for 2.5 hours at a temperature of 48°C to 55°C. The resulting polymer dispersion had a Brookfield viscosity of 10000cps. To the above dispersion was added 10 grams of 99% acetic acid and 20 grams of sodium sulfate. The resulting dispersion had a Brookfield viscosity of 2825 cps and contained 20% of a 95/5 copolymer of acrylamide and DMAEA-MCQ with an intrinsic viscosity of 14.1 dl/gm in 0.125 molar NaNO₃.

Example 3 - Process for Synthesizing Dispersion Copolymers of Acrylamide and 10 mole % DMAEA-MCQ.

[0025] To a two-liter resin reactor equipped with stirrer, temperature controller, and water cooled condenser, was added 239.38 grams of a 48.1% solution of acrylamide (1.6199 moles), 21.63 grams of an 80.6% solution of DMAEA-MCQ (0.09001 moles), 260 grams of ammonium sulfate. 258.01 grams of deionized water, 18 grams of glycerol, 33.75 grams of a 16% solution of polyDADMAC (IV= 1.5 dl/gm), 36 grams of a 20% solution of polyDMAEA-MCQ (IV=2.0dl/gm), and 0.3 grams of EDTA. The mixture was heated to 48°C and 0.50 grams of a 4% solution of 2,2' Azobis(2 amidinopropane) dihydrochloride was added. The resulting solution was sparged with 1000 cc/min. of nitrogen. After 15 minutes, polymerization began and the solution became viscous. Over the next 4 hours the temperature was maintained at 48°C and a solution containing 79.79 grams (0.5399 moles) of 48.1% acrylamide, 36.04 grams (0.1500 moles) of an 80.6% solution of DMAEA-MCQ, 6 grams of glycerol and 0.1 gram of EDTA was pumped into the reactor using a syringe pump. To the resulting polymer dispersion was added 0.50 grams of a 4% solution of 2,2' Azobis(2 amidinopropane) dihydrochloride. The dispersion was then further reacted for 2.5 hours at a temperature of 48°C to 55°C. The resulting polymer dispersion had a Brookfield viscosity of 7600cps. To the above dispersion was added 10 grams of 99% acetic acid and 20 grams of sodium sulfate. The resulting dispersion had a Brookfield viscosity of 2100 cps and contained 20% of a 90/10 copolymer of acrylamide and DMAEA-MCQ with an intrinsic viscosity of 15.5 dl/gm in 0.125 molar NaNO₃.

Example 4 - Process for Synthesizing Dispersion Copolymers of Acrylamide and 20 mole % DMAEA-MCQ.

[0026] To a two-liter resin reactor equipped with stirrer, temperature controller, and water cooled condenser was added 136.03 grams of a 48.1 % solution of acrylamide (0.9205 moles), 37.12 grams of an 80.6% solution of DMAEA-MCQ (0.1545 moles), 190 grams of ammonium sulfate, 50 grams of sodium sulfate, 267.99 grams of deionized water, 13.2 grams of glycerol, 33.75 grams of a 16% solution of polyDADMAC (IV= 1.5 dl/gm), 45 grams of a 20% solution of polyDMAEA-MCQ (IV=2.0dl/gm), and 0.2 grams of EDTA. The mixture was heated to 48°C and 0.50 grams of a 4% solution of 2,2' Azobis (2 amidinopropane) dihydrochloride was added. The resulting solution was sparged with 1000 cc/min. of nitrogen. After 15 minutes, polymerization began and the solution became viscous. Over the next 4 hours the temperature was maintained at 48°C and a solution containing 111.29 grams of 48.1% acrylamide, 63.47 grams (0.2641 moles) of an 80.6% solution of DMAEA-MCQ, 10.8 grams of glycerol and 0.2 grams of EDTA was pumped into the reactor using a syringe pump. To the resulting polymer dispersion was added 0.50 grams of a 4% solution of 2,2' Azobis (2 amidinopropane) dihydrochloride. The dispersion was then further reacted for 2.5 hours at a temperature of 48°C to 55°C. The resulting polymer dispersion had a Brookfield viscosity of 2160 cps. To the above dispersion was added 10 grams of 99% adipic acid and 30 grams of ammonium sulfate. The resulting dispersion had a Brookfield viscosity of 1325 cps and contained 20% of an 80/20 copolymer of acrylamide and DMAEA-MCO with an intrinsic viscosity 13.7 dl/gm in 0.125 molar NaNO₃.

[0027] The following examples utilized the test polymers and furnishes described below.

Polymer Samples**POLYMER DESCRIPTION**

[0028]

EP 0 831 177 B1

Dispersions

5	Dispersion A	3 mole% DMAEA•MCQ	IV 12.1 dUg
	Dispersion B	5 mole% DMAEA•MCQ	IV 14.1 dUg
	Dispersion C	10 mole % DMAEA•MCQ	IV 14.8 dl/g
	Dispersion D	10 mole% DMAEA•MCQ	IV 17.0 dl/g
	Dispersion E	10 mole% DMAEA•MCQ	IV 18.2 dl/g
	Dispersion F	20 mole% DMAEA•MCQ	IV 21.2 dl/g
10	Dispersion G	20 mole% DMAEA•MCQ	IV 19.4 dUg
	Dispersion H	10 mole% DMAEA•MCQ	IV 19.2 dl/g

Other Polymers

	Polymer A	10 mole% DMAEA•MCQ Latex	IV 17.7 dl/g
15	Polymer B	10 mole% DMAEA•MCQ Latex	IV 19.1 dl/g
	Polymer C	10 mole% DMAEA•BCQ Dispersion	IV 12.9 dl/g
	Polymer D	70/30 mole% AcAm/NaAc Latex	
	Polymer E	10 mole% DMAEA•MCQ Dry polymer (Floerger)	
	Polymer F	Epi-DMA solution polymer	
20	Polymer G	Poly(DADMAC) solution polymer	IV 0.55 dl/g
	Polymer H	Poly(DADMAC) solution polymer	IV 1.9 dl/g

IV Measurement

- 25 **[0029]** IV measurements of polymer samples were carried out in 0.125 M NaNO₃ solution. The procedure comprises:
1. Prepare a 1% dispersion product (0.2% polymer actives) solution by injecting 2 g of the dispersion polymer with a syringe into the vortex of 198 g of DI water. Continue stirring at 800 rpm for 30 minutes.
 2. Prepare a 0.045% polymer actives working solution from:

30	0.2% Polymer actives solution	22.5 g
	Sodium acetate solution	1.0 g
	0.25 Molar Sodium nitrate	50.0 g
	DI water	26.5 g

- 35
3. Fill 2 ml of 0.125 Molar sodium nitrate solution into a capillary viscometer. Measure the time t_s .
 4. Remove the sodium nitrate solution and clean the viscometer. Fill 2 ml of the 0.045% polymer actives solution into the viscometer. Measure the time t_1 .

Furnish Preparation

- 40 **[0030]** Three of the furnishes used for polymer activity testing were prepared from thick stock obtained from paper mills and diluted to a consistency of approximately 0.5% with formulation water. The fourth furnish was a synthetic alkaline furnish which comprised 70 weight % fiber and 30 weight % filler, diluted to a consistency of approximately 0.5%
- 45 with formulation water. The formulation water contained 200 ppm calcium hardness (added as CaCl₂), 152 ppm magnesium hardness (added as MgSO₄) and 110 ppm bicarbonate alkalinity (added as NaHCO₃).

Drainage and Retention Tests

- 50 **[0031]** The Britt CF Dynamic Drainage jar was used for uniform mixing of polymer and furnish; the mixing speed of the Britt jar was 500 rpm. The drainage tester simulates gravity drainage on a paper machine. The test procedures for drainage and retention are given below:

1. Measure a 500 ml sample of the thin stock using a graduated cylinder.
 2. Add thin stock to the Britt jar.
 3. Begin stirring (500 rpm) and add starch or coagulant using a syringe (when required).
 4. After 10 seconds, add polymer solution to the furnish using a syringe.
 5. Stop stirring after a total time of 30 seconds. i.e. 20 seconds after adding polymer.
- 55

6. Immediately transfer the treated furnish into the reservoir of the drainage tester.
7. Remove the stopper and collect the filtrate for 5 seconds.
8. Record the weight of filtrate.
9. Measure the filtrate turbidity at 450 nm on a DR-2000 Spectrometer. The filtrate was diluted (x2) with DI water.

5
[0032] Drainage and turbidity data were obtained for dispersion and latex polymers using the test procedures described above. In these examples, a measure of retention is given by the percent reduction in the turbidity obtained with no polymer treatment (blank). Dosage curves of Drainage Improvement (%) and Turbidity Reduction (%) were determined for polymers tested. It is well known that the retention and drainage activities of polymers depend on several factors including the type of furnish to be treated. For this reason furnishes were selected which were significantly different from each other. The first was a 100% recycled linerboard furnish. The second was a furnish used for the production of corrugated folding grade products. This furnish was a mixture of old corrugated cardboard (OCC), newsprint and box-board. Thick stocks and other additives used for the manufacture of publication grade paper were collected to prepare the third furnish. The fourth furnish was prepared in the laboratory and closely resembles the alkaline furnish used by the paper industry for the production of fine paper.

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[0033] Using the test furnishes described above, the Drainage and Retention tests also described above were employed to determine drainage and retention activities of Dispersions A-H and Polymers A-H in Examples 5-9.

20 Example 5

[0034] The initial activity testings of the DMAEA-MCQ dispersion polymers were done with 100% recycled linerboard. This furnish contained no added filler and retention was primarily for fines from the fiber. Figure 1 shows a plot of % turbidity reduction vs polymer dosage for three of the hydrophilic dispersion polymers and Polymer A, a standard latex flocculant. The compositions of the dispersions were (1) AcAm/DMAEA•MCQ:97/3, (2) AcAm/DMAEA•MCQ:95/5, and (3) AcAm/DMAEA•MCQ:90/10. Dispersions A, B and C showed increased efficiency of retention performance as compared to Polymer A. In addition, Figure 1 shows that turbidity reductions between 60 and 70% were achieved with the dispersion polymers for dosage of approximately 0.4 kg active / 1000 kg (0.8 lbs active/t).

25
30
[0035] Figure 2 shows the drainage improvements realized by the dispersion polymers described above. The copolymer containing 5 mole % DMAEA-MCQ showed the best drainage behavior amongst the dispersions. However, the latex polymer, Polymer A, outperformed the dispersions for the entire dosage range tested. It should be noted that the intrinsic viscosities of the first batches of hydrophilic dispersions were significantly lower than Polymer A.

35 Example 6

[0036] The corrugated coated furnish was a mixture of OCC, newsprint and boxboard. Unlike the recycled linerboard this furnish contained CaCO₃ as filler. The % ash was found by gravimetric measurement to be 7.3%. Preliminary activity testings were carried out with the lower IV (11.9 - 15.7 dl/g) polymer samples and the data indicated some important trends in polymer performances. Both retention and drainage performances of the dispersion polymers improved with increasing mole% of DMAEA•MCQ. Overall, the 10 mole% DMAEA-MCQ copolymer showed the best drainage and retention performances among the dispersions tested.

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[0037] The retention performances of the higher IV (17.0 - 21.2 dl/g) dispersion copolymers containing 10 and 20 mole% DMAEA•MCQ are shown in Figure 3. Dispersions D, E, F and G, containing 10 and 20 mole%. DMAEA•MCQ showed comparable retention activities to Polymer A with corrugated coated furnish.

45
[0038] Figure 4 shows the drainage activities of the higher IV dispersion copolymers containing 10 and 20 mole% DMAEA•MCQ. The results clearly demonstrate that for the dosage range 0 to 0.7 kg active/1000 kg (0 to 1.5 lbs active/t) the hydrophilic dispersion polymers were comparable to the standard flocculant, Polymer A. As the polymer dosage was increased to 1.8 kg active / 1000 kg (4.0 lbs active/t.), the 20 mole% DMAEA•MCQ copolymers continued to show drainage behavior similar to Polymer A.

50 Example 7

[0039] The publication grade furnish was a blend of 90% (softwood, hardwood, high ash broke, low ash broke) and 10% (CaCO₃, TiO₂, starch, alum). The flocculant used at the time of the test was Polymer D (AcAm/NaAc:70/30). Figure 5 shows the results of Britt jar screening of dispersion and dry polymers. On an equal actives basis at 0.7 kg/1000 kg (1.5 lbs/t), the 10 mole% DMAEA•MCQ dispersion (Dispersion E) outperformed all polymers including Polymer C, Polymer D and Polymer E, a dry polymer available from Floereger.

55
[0040] Results of retention and drainage testings performed with this furnish are given in Figures 6 and 7. Two hydrophilic dispersions containing 10 mole% DMAEA•MCQ were compared with Polymer A and Polymer D. The plot of %

turbidity reduction vs dosage, Figure 6, shows that for low dosages of flocculants significant reductions in turbidity (~90%) were achieved for each polymer. In addition, there were no differences in retention activities among the dispersion and latex polymers.

[0041] Figure 7 shows that the drainage activities of the latex and dispersion polymers were quite different. The latex polymer Polymer A, gave the best drainage performance. This was followed by the higher IV dispersion polymer. At dosages above 0.45 kg/1000kg (1.0 lb/t), the drainage improvements for the two dispersion polymers were greater than Polymer D.

Example 8

[0042] A synthetic alkaline furnish was prepared, containing approximately 30% CaCO₃ as filler and, therefore, had the highest filler loading among the furnishes prepared. In Example 8, cationic starch was charged to the furnish in the amount of about 4.5 kg/1000 kg (10 lb/ton) of dry weight of slurry solids.

[0043] Figure 8 shows the dosage retention curves for two hydrophilic dispersions containing 10 mole% and 20 mole% DMAEA-MCQ (Dispersion E, G) compared to Polymer B and Polymer C. Polymer B (IV 19.1 dl/g) is a higher molecular weight material than Polymer A (IV 17.7 dl/g). The results indicate that the hydrophilic dispersion polymers containing 10 and 20 mole% DMAEA-MCQ are also very effective retention aids for fine paper application.

[0044] Drainage data for the polymers tested with the standard alkaline furnish are given in Figure 9. The hydrophilic dispersion containing 20 mole% DMAEA-MCQ showed better drainage than Polymer C and the 10 mole% DMAEA-MCQ dispersion polymer. Its drainage performance was comparable to Polymer B.

[0045] The preceding results demonstrated that the hydrophilic dispersion polymers are effective retention and drainage aids for a range of furnishes. The activities of the new dispersion polymers in the single polymer program were comparable to or sometimes better than the inverse emulsion polymer, Polymer A

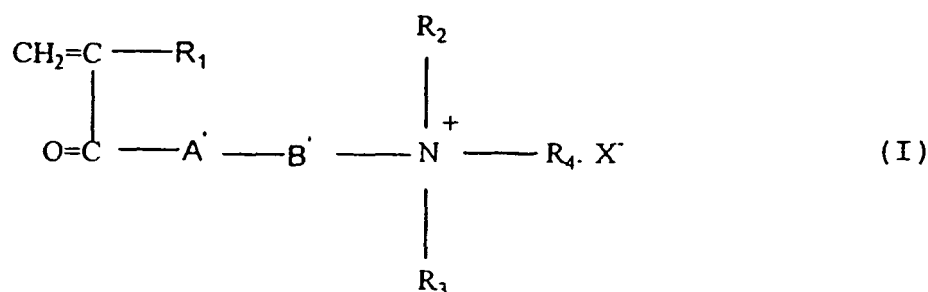
Example 9

[0046] The effects of coagulants on the retention and drainage activities of two DMAEA-MCQ dispersion polymers (10 and 20 mole% DMAEA-MCQ) were evaluated in a dual polymer program and are shown in Figures 10 and 11. The corrugated coated furnish was selected for this study. Coagulants, including Polymer F, (Epi/DMA), Polymer G (polyDADMAC IV = 0.55) and Polymer H (polyDADMAC, IV = 1.9) were used. Figure 10 shows an increase of approximately 30% in retention performance for Dispersion F, the 20 mole% DMAEA-MCQ polymer with the addition of 0.9 kg/1000 kg (2.0 lbs/ton) of the high IV polyDADMAC. There were also measurable increases in retention with Polymer F and Polymer G. There were no significant changes in retention activities for the 10 mole% DMAEA-MCQ (Dispersion D) polymer with the addition of coagulants. The coagulants showed a less beneficial effect on the drainage activities of the two DMAEA-MCQ dispersion polymers (Figure 11).

Claims

1. Use of a hydrophilic dispersion polymer resulting from the polymerization of monomers comprising:

(i) a cationic monomer of the formula (I)



wherein R₁ is H or CH₃; each of R₂ and R₃ is an alkyl group having 1 to 2 carbon atoms; R₄ is H or an alkyl group of 1 or 2 carbon atoms; A' is an oxygen atom or NH; B' is an alkylene group of 2 to 4 carbon atoms or a hydroxypropylene group; and X⁻ is an anionic counterion; and

(ii) a second monomer represented by (meth)acrylamide in an aqueous solution of a polyvalent anionic salt to increase retention and/or drainage in a papermaking process comprising:

- a) forming an aqueous cellulosic papermaking slurry;
- b) adding an effective amount of said hydrophilic dispersion polymer to the slurry;
- c) draining the slurry to form a sheet; and
- d) drying the sheet;

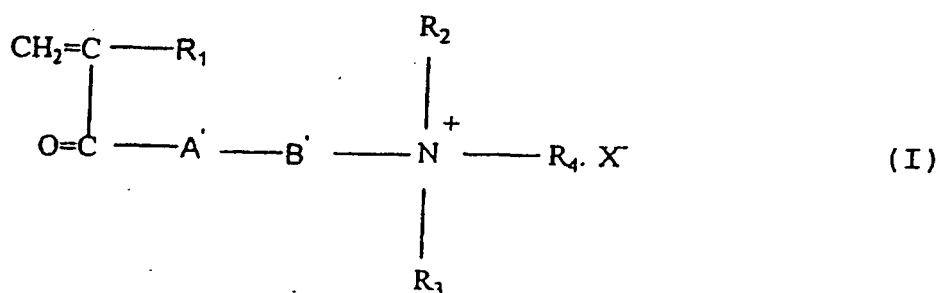
provided that the hydrophilic dispersion polymer is not used for the treatment of recycled coated broke.

2. The use of claim 1 wherein the hydrophilic dispersion polymer is a copolymer of dimethylaminoethyl acrylate methyl chloride quaternary and acrylamide and wherein the amount of dimethylaminoethyl acrylate methyl chloride quaternary present in the copolymer is from about 2 mol% to about 20 mol%.
3. The use of claim 1 wherein the dispersion polymer is added in an amount of from about 0.4 to about 0.9 kg (0.8 to about 2.0 pounds) of active per 1000 kg of slurry solids.
4. The use of claim 1 wherein the hydrophilic dispersion polymer has an intrinsic viscosity of from about 11.9 to about 21.2 deciliters per gram.
5. The use of claim 1 further comprising the step of adding a coagulant before the addition of the hydrophilic dispersion polymer, the coagulant being selected from the group consisting of Epi/DMA and polymeric diallyl dimethyl ammonium chloride.
6. The use of claim 5 further comprising the step of adding a cationic starch to the furnish in an amount of about 4.5 kg/1000 kg (10 lb/ton) of dry weight of slurry solids.

Patentansprüche

1. Verwendung eines hydrophilen Dispersions-Polymers, welches aus der Polymerisation von Monomeren, welche umfassen:

(i) ein kationisches Monomer der Formel (I)



worin R₁ für H oder CH₃ steht; R₂ und R₃ jeweils eine Alkylgruppe mit 1 bis 2 Kohlenstoffatomen sind; R₄ für H oder eine Alkylgruppe mit 1 oder 2 Kohlenstoffatomen steht; A' ein Sauerstoffatom oder NH ist; B' eine Alkylgruppe mit 2 bis 4 Kohlenstoffatomen oder eine Hydroxypropylengruppe ist; und X⁻ ein anionisches Gegenion ist; und

(ii) ein zweites Monomer, das durch (Meth)acrylamid dargestellt wird, in einer wässrigen Lösung eines mehrwertigen anionischen Salzes resultiert, um die Zurückhaltung und/oder das Ablauflassen in einem Papierherstellungsverfahren zu erhöhen, welches umfasst:

- a) Bilden einer wässrigen Cellulose-haltigen Papierherstellungsaufschlämmung;
- b) Zugabe einer wirksamen Menge des hydrophilen Dispersionspolymers zu der Aufschlämmung;
- c) Ablauflassen der Aufschlämmung, um ein Blatt zu bilden; und

d) Trocken des Blatts;

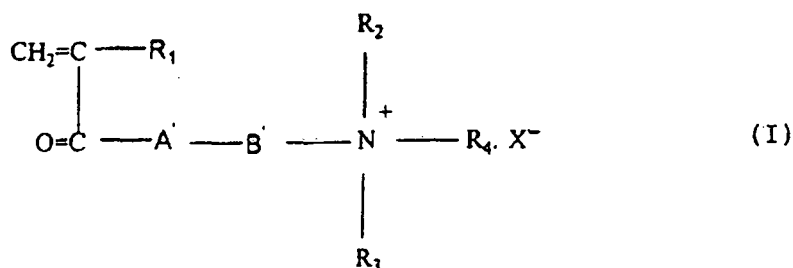
vorausgesetzt, dass das hydrophile Dispersionspolymer nicht für die Behandlung von zurückgeführtem beschichtetem Ausschuss verwendet wird.

- 5
2. Verwendung nach Anspruch 1, bei dem das hydrophile Dispersionspolymer ein Copolymer von quaternärem Dimethylaminoethylacrylatmethylchlorid und Acrylamid ist und wobei die Menge an quaternärem Dimethylaminoethylacrylatmethylchlorid, die in dem Copolymer vorliegt, etwa 2 Molprozent bis etwa 20 Molprozent beträgt.
- 10
3. Verwendung nach Anspruch 1, bei der das Dispersionspolymer in einer Menge von etwa 0,4 bis etwa 0,9 kg (0,8 bis etwa 2,0 Pfund) an aktivem Material pro 1000 kg Aufschlammungsfeststoffe zugesetzt wird.
- 15
4. Verwendung nach Anspruch 1, bei der das hydrophile Dispersionspolymer eine innere Viskosität von etwa 11,9 bis etwa 21,2 Deziliter pro Gramm aufweist.
- 20
5. Verwendung nach Anspruch 1, weiter umfassend den Schritt der Zugabe eines Koagulans vor der Zugabe des hydrophilen Dispersionspolymers, wobei das Koagulans ausgewählt ist aus der Gruppe bestehend aus Epi/DMA und polymerem Diallyldimethylammoniumchlorid.
6. Verwendung nach Anspruch 5, weiter umfassend den Schritt der Zugabe einer kationischen Stärke zu den Papierrohstoffen in einer Menge von 4,5 kg/1000 kg (10 lb/Tonne) Trockengewicht an Aufschlammungsfeststoffen.

25 **Revendications**

1. Utilisation d'un polymère à dispersion hydrophile résultant de la polymérisation de monomères comprenant :

(i) un monomère cationique de formule (I)



dans laquelle R₁ est un H ou un CH₃ ; chacun des R₂ et R₃ est un groupe alkyle possédant 1 à 2 atomes de carbone ; R₄ est un H ou un groupe alkyle possédant 1 à 2 atomes de carbone ; A' est un atome d'oxygène ou NH ; B' est un groupe alkylène de 2 à 4 atomes de carbone ou un groupe hydroxypropylène ; et X⁻ est un contre-ion anionique ; et

45 (ii) un second monomère représenté par un méth(acrylamide) dans une solution aqueuse d'un sel anionique polyvalent pour augmenter la rétention et/ou l'égouttage dans un procédé de fabrication du papier comprenant les étapes consistant à:

- 50
- a) former une suspension pâteuse cellulosique en phase aqueuse ;
 - b) ajouter une quantité effective dudit polymère à dispersion hydrophile à la suspension pâteuse ;
 - c) égoutter la suspension pâteuse pour former une feuille ; et
 - d) sécher la feuille ;

55 à condition que le polymère à dispersion hydrophile ne soit pas utilisé pour le traitement de cassés de fabrication enrobés, recyclés.

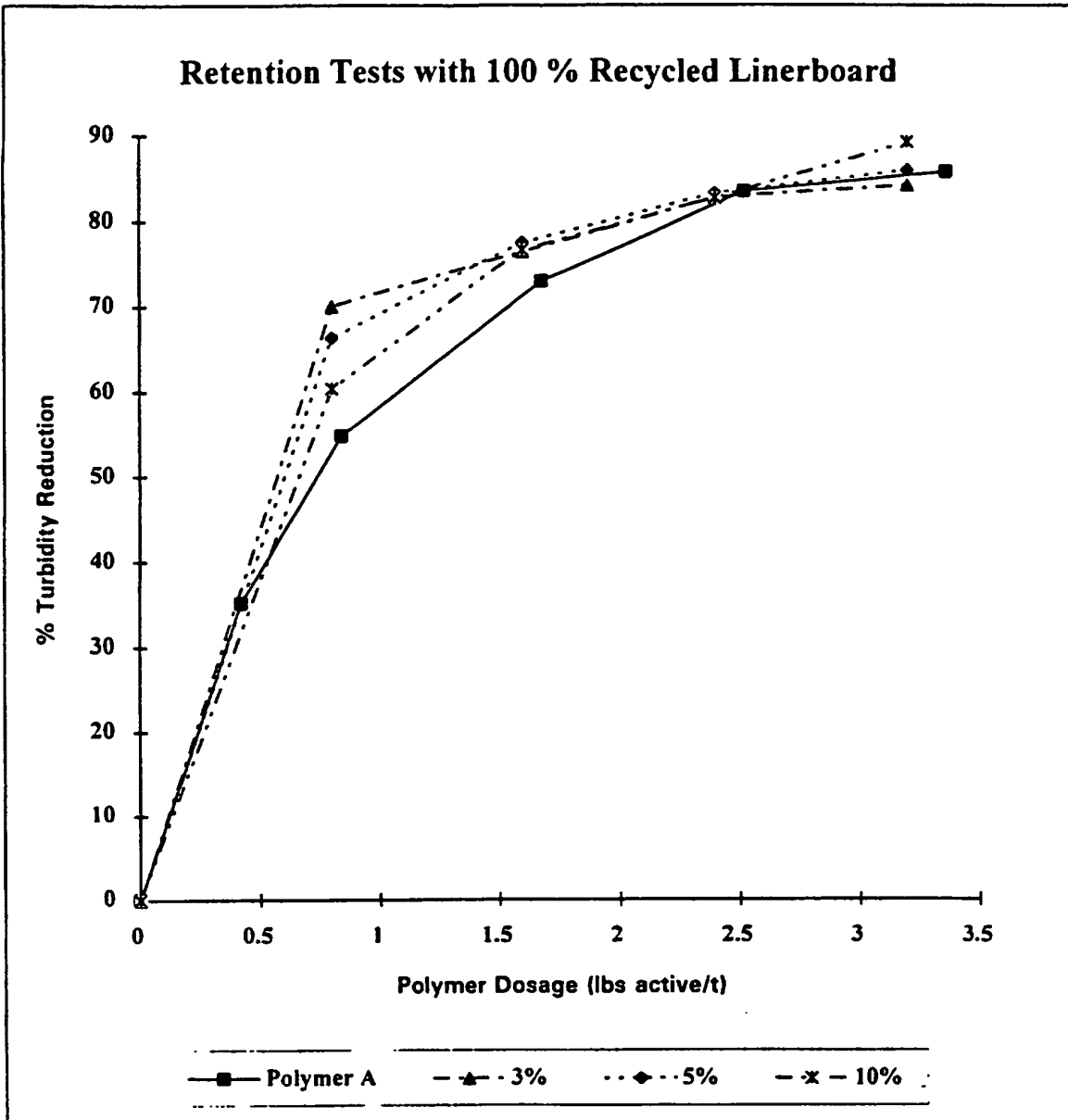
2. Utilisation selon la revendication 1 dans laquelle le polymère à dispersion hydrophile est un copolymère d'acrylate de diméthylaminoéthyle quaternisé avec du chlorure de méthyle et d'acrylamide et dans laquelle la quantité d'acrylate

EP 0 831 177 B1

de diméthylaminoéthyle quaternisé avec du chlorure de méthyle présent dans le copolymère est située entre environ 2 % en mole et environ 20 % en mole.

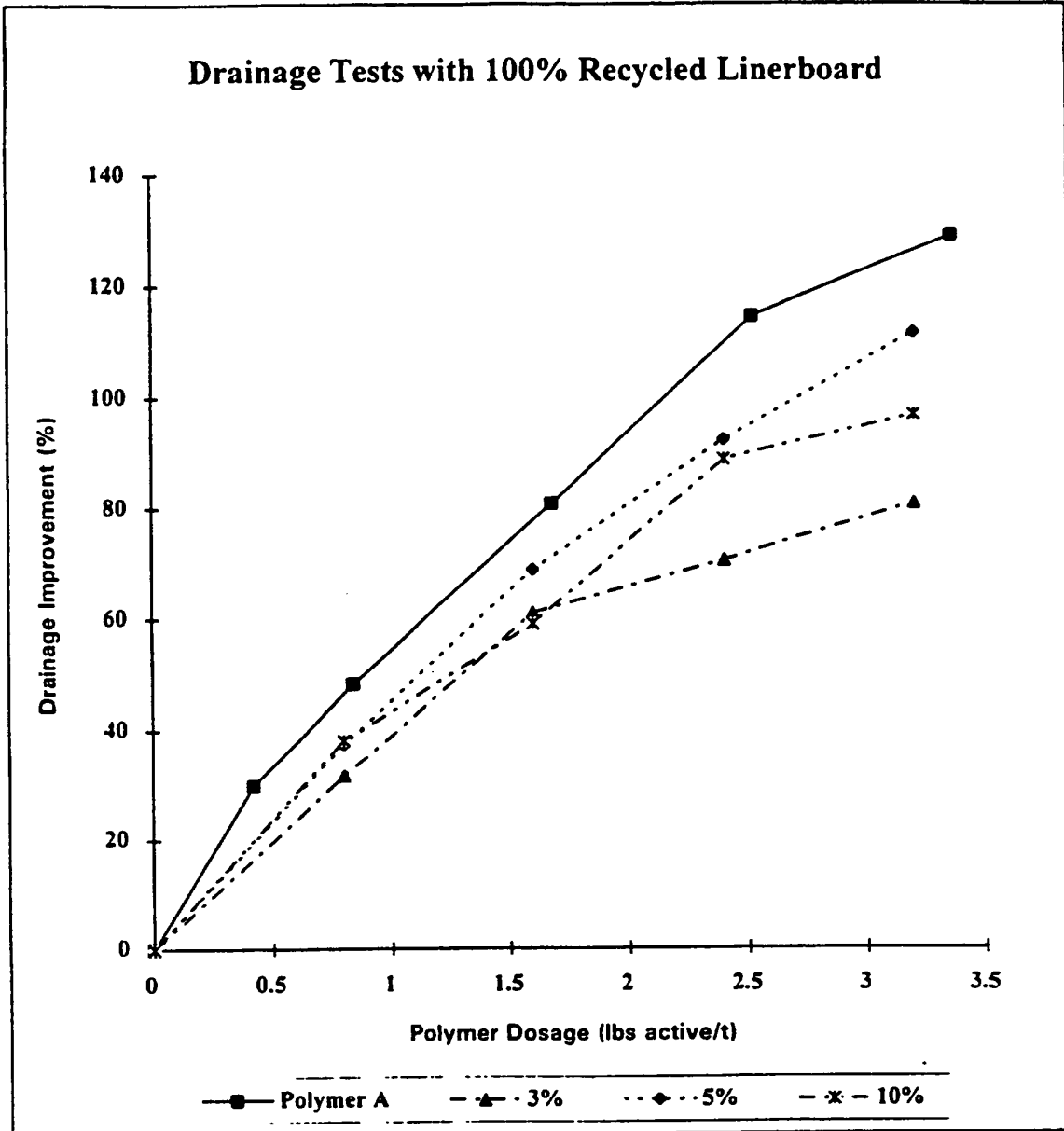
- 5
3. Utilisation selon la revendication 1 dans laquelle le polymère à dispersion est ajouté en une quantité située entre environ 0,4 et environ 0,9 kg (0,8 à environ 2,0 livres) de matière active par 1000 kg de matière sèche de la suspension pâteuse.
- 10
4. Utilisation selon la revendication 1 dans laquelle le polymère à dispersion hydrophile possède une viscosité intrinsèque entre environ 11,9 et environ 21,2 décilitres par gramme.
- 15
5. Utilisation selon la revendication 1 comprenant en outre l'étape consistant à ajouter un coagulant avant l'ajout du polymère à dispersion hydrophile, le coagulant étant sélectionné dans le groupe comprenant l'Epi/DMA et le chlorure d'ammonium de diallyldiméthyle polymérique.
- 20
6. Utilisation selon la revendication 5 comprenant en outre l'étape consistant à ajouter une urée cationique à la composition de fabrication en une quantité d'environ 4,5 kg/1000 kg (10 livres/tonne) de poids sec de matière sèche de la suspension pâteuse.
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FIGURE 1



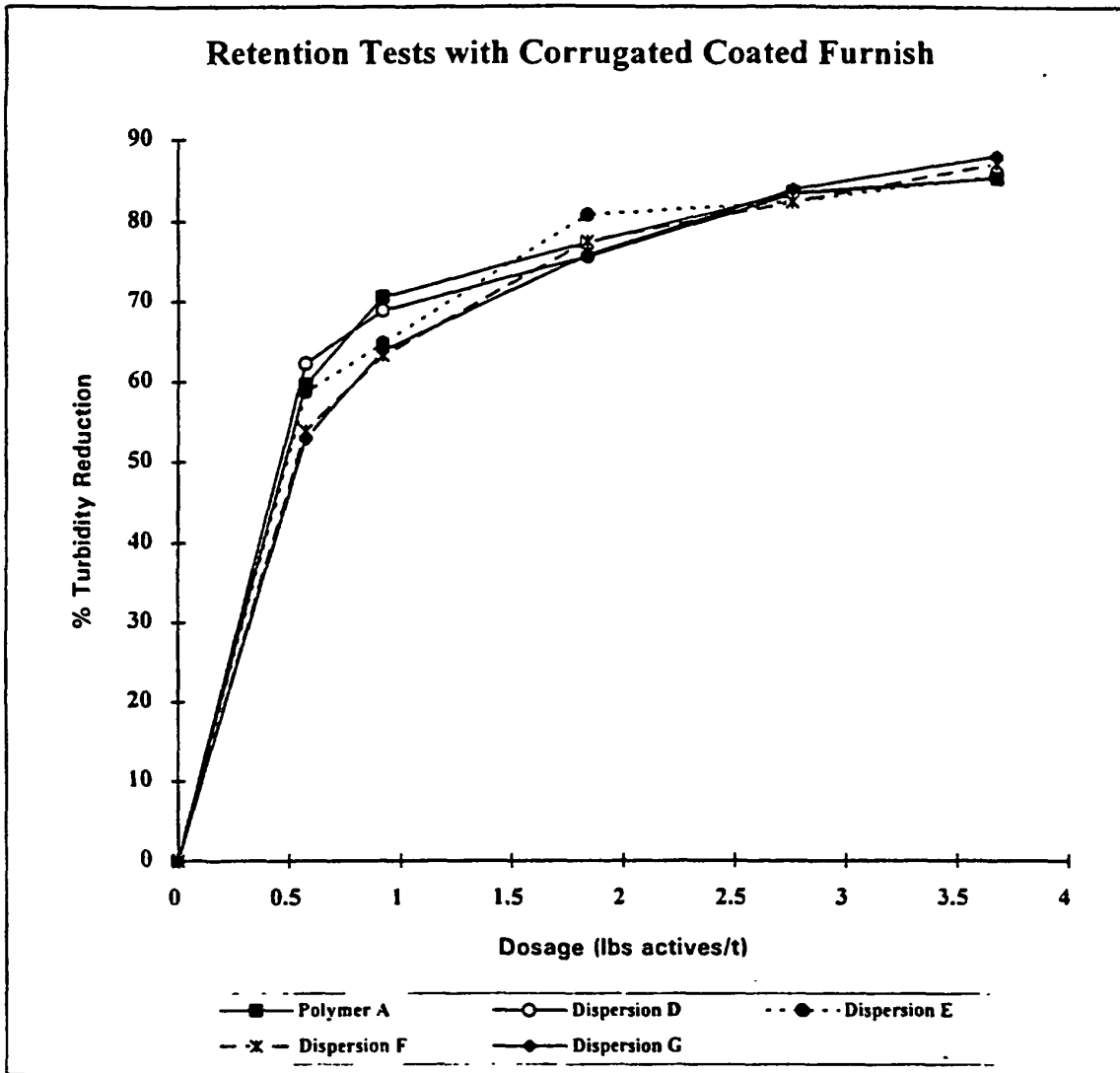
Polymer Description	Cationic Mole %	DMAEA. MCQ Mole %	DMAEA. BCQ Mole %	IV 0.125N NO3 (dl/g)
Polymer A	10	10	0	17.7
Dispersion A	3	3	0	12.1
Dispersion B	5	5	0	14.1
Dispersion C	10	10	0	14.8

FIGURE 2



Polymer Description	Cationic Mole %	DMAEA. MCQ Mole %	DMAEA. BCQ Mole %	IV 0.125N NO3 (dl/g)
Polymer A	10	10	0	17.7
Dispersion A	3	3	0	12.1
Dispersion B	5	5	0	14.1
Dispersion C	10	10	0	14.8

FIGURE 3



Polymer Description	Cationic Mole %	DMAEA. MCQ Mole %	DMAEA BCQ Mole %	IV 0.125N NO3 (dl/g)
Polymer A	10	10	0	17.70
Dispersion D	10	10	0	17.00
Dispersion E	10	10	0	18.20
Dispersion F	20	20	0	21.20
Dispersion G	20	20	0	19.40

FIGURE 4

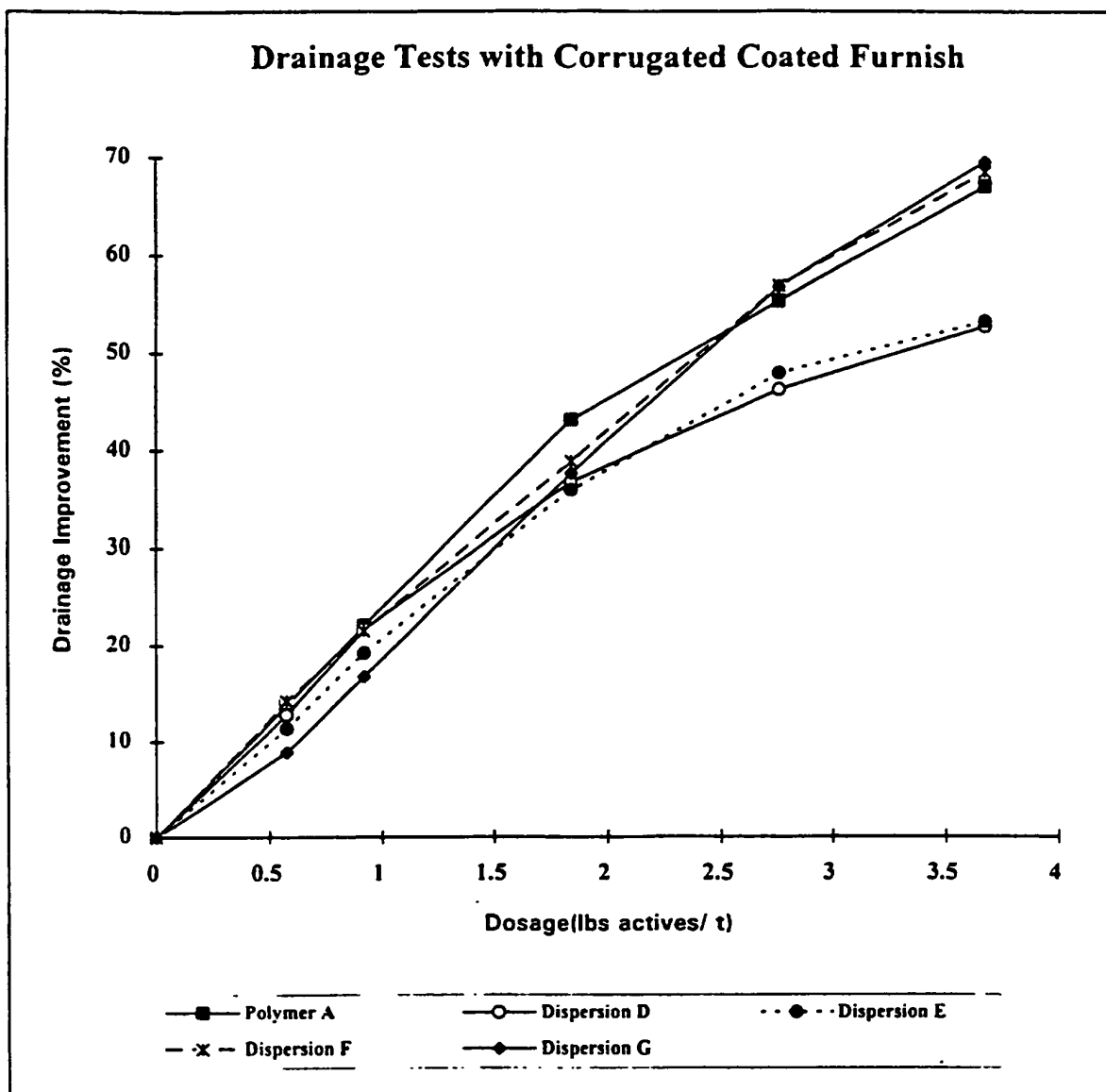


FIGURE 5
Retention Tests with Publication Grade Furnish

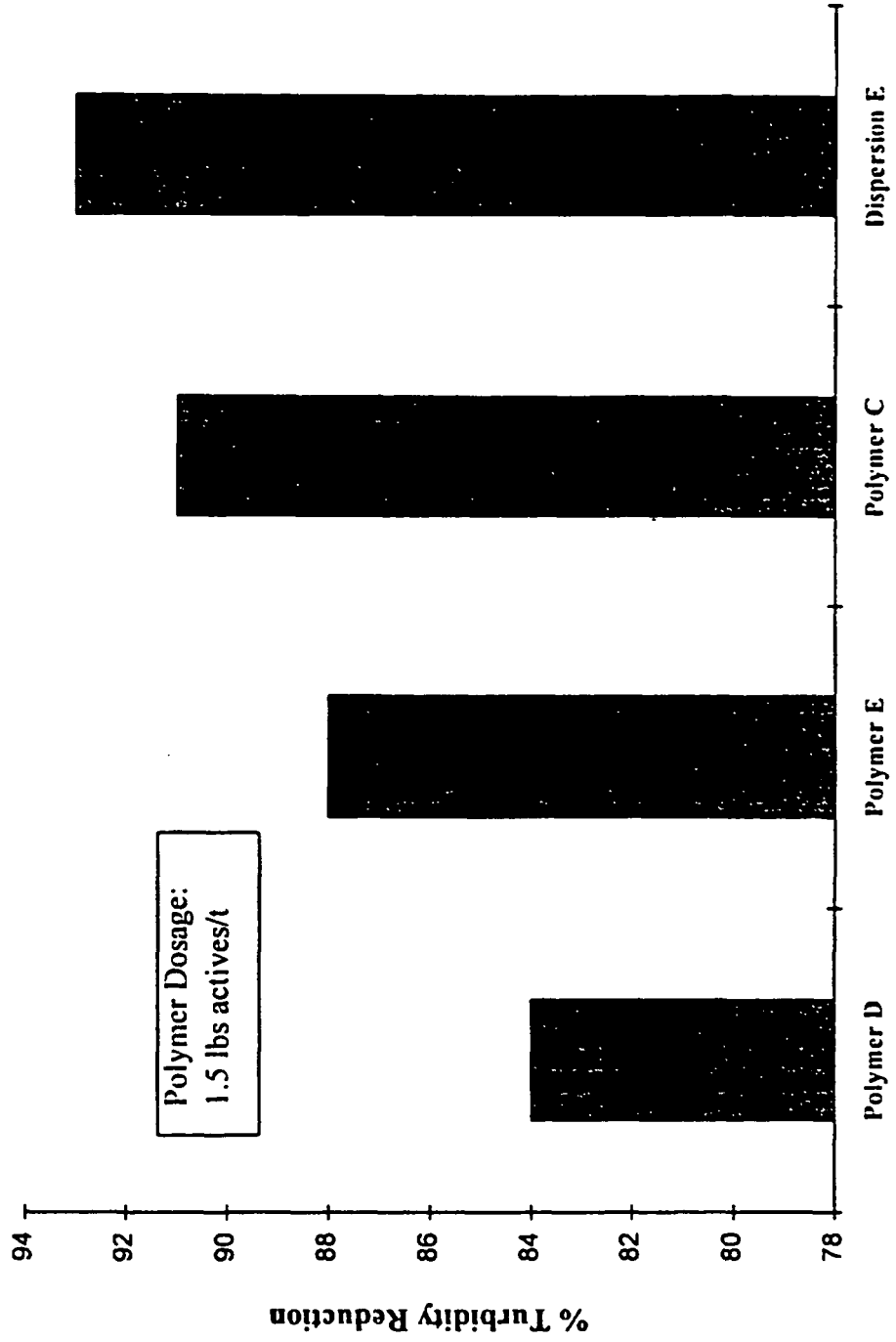
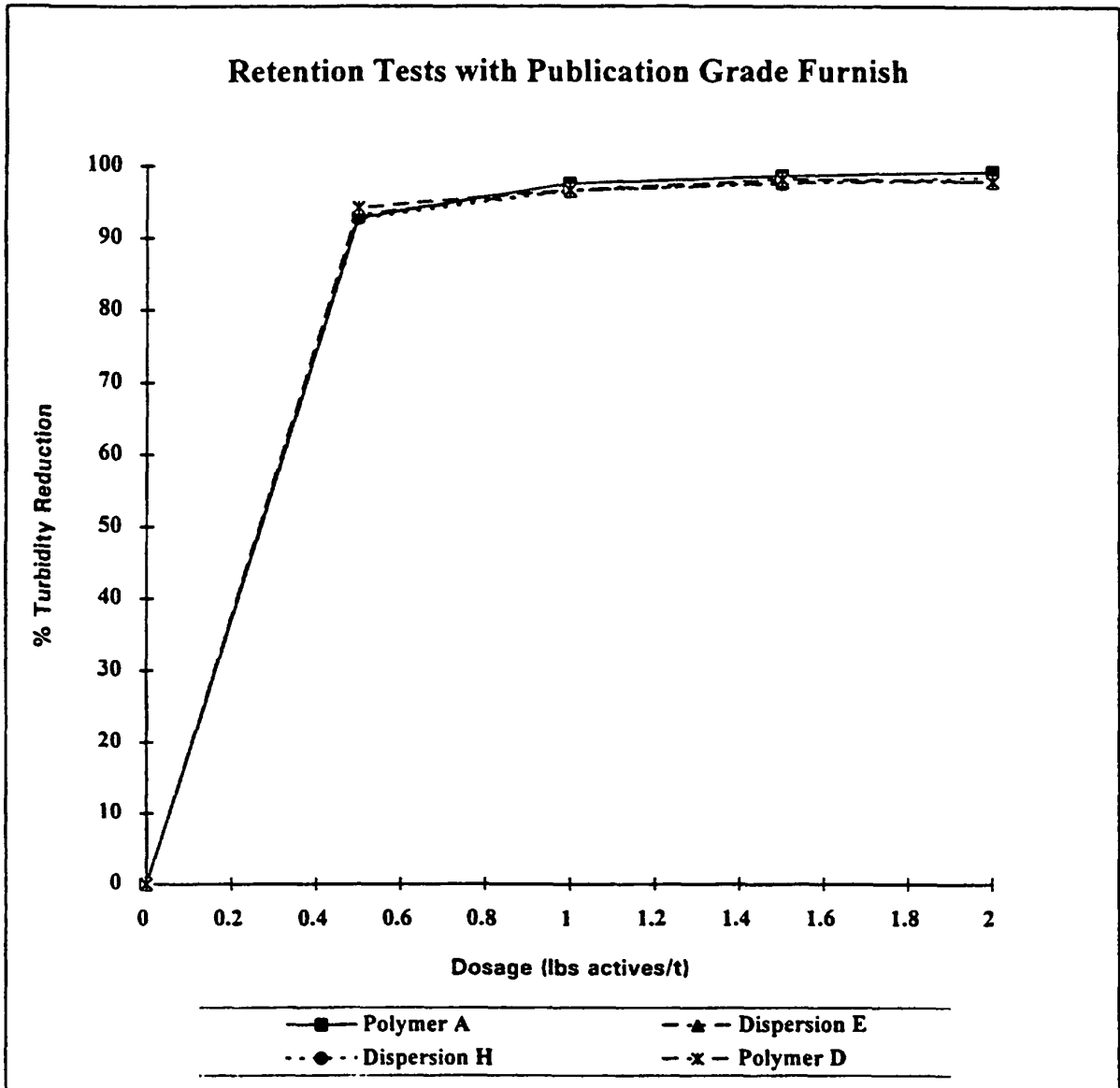
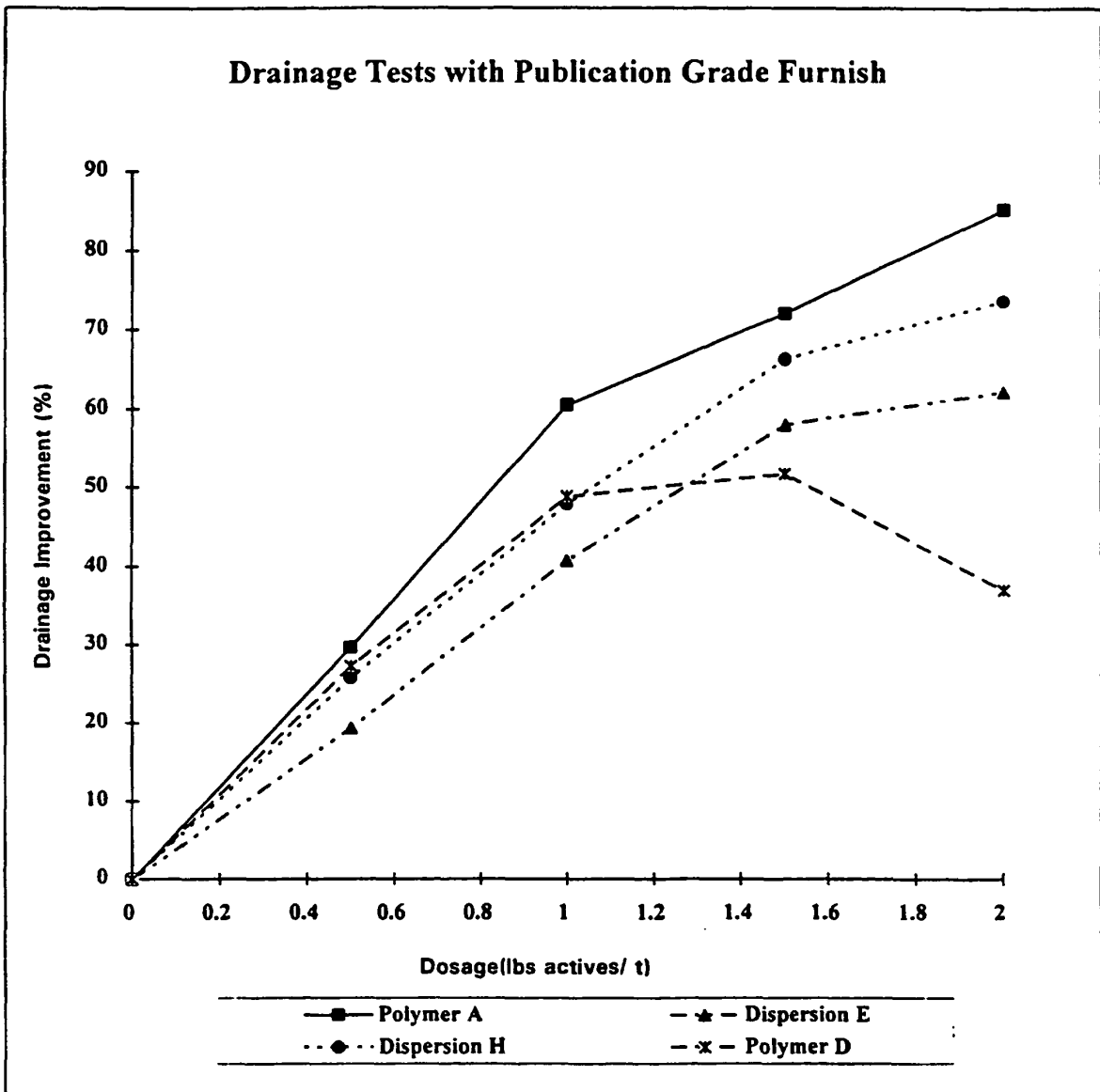


FIGURE 6



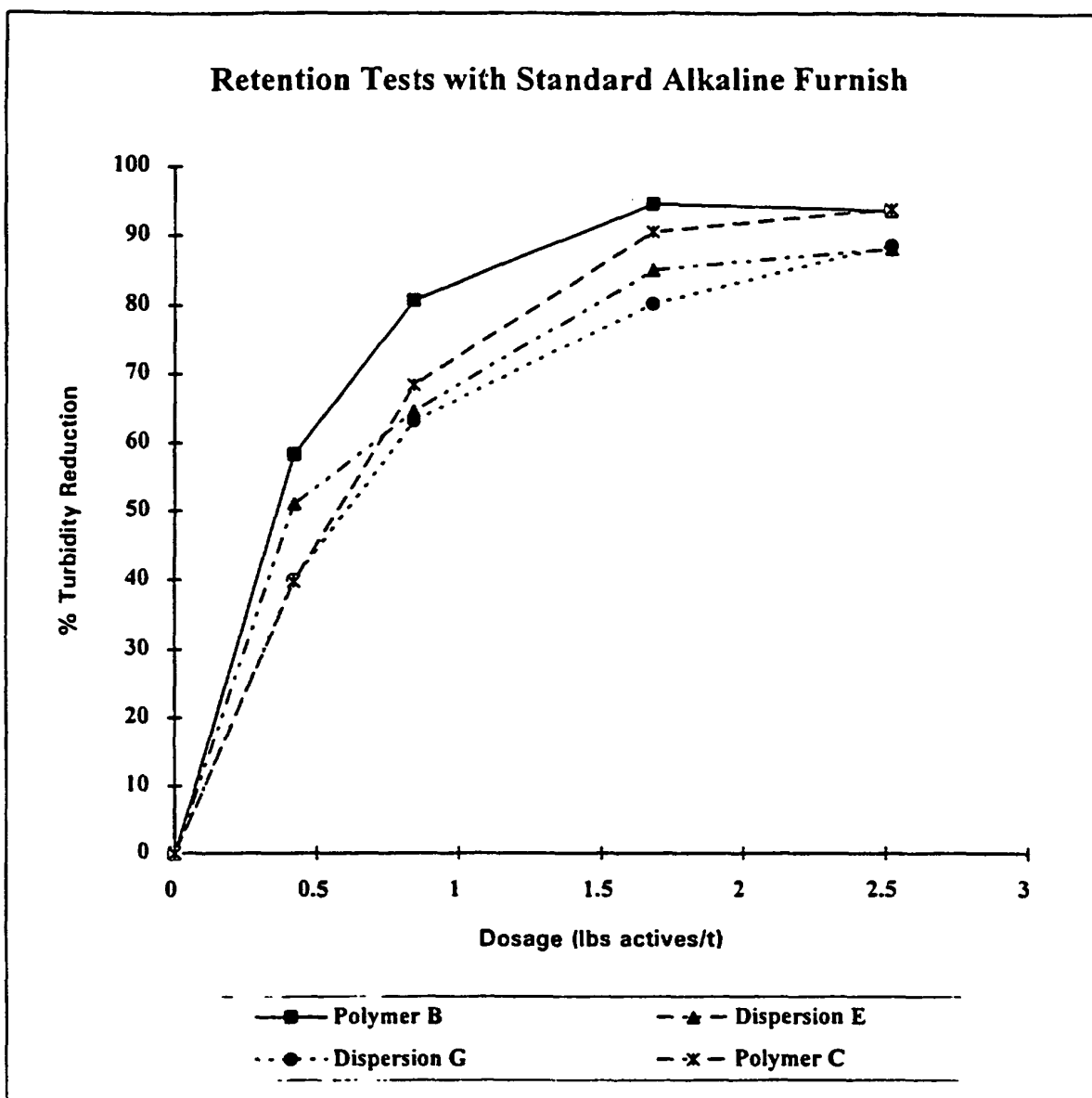
Polymer Description	Cationic Mole %	DMAEA. MCQ Mole %	DMAEA BCQ Mole %	IV 0.125N NO3 (dl/g)
Polymer A	10	10	0	17.7
Dispersion E	10	10	0	18.2
Dispersion H	10	10	0	19.2
Polymer D	70/30 mole% AcAm/NaAc		-	-

FIGURE 7



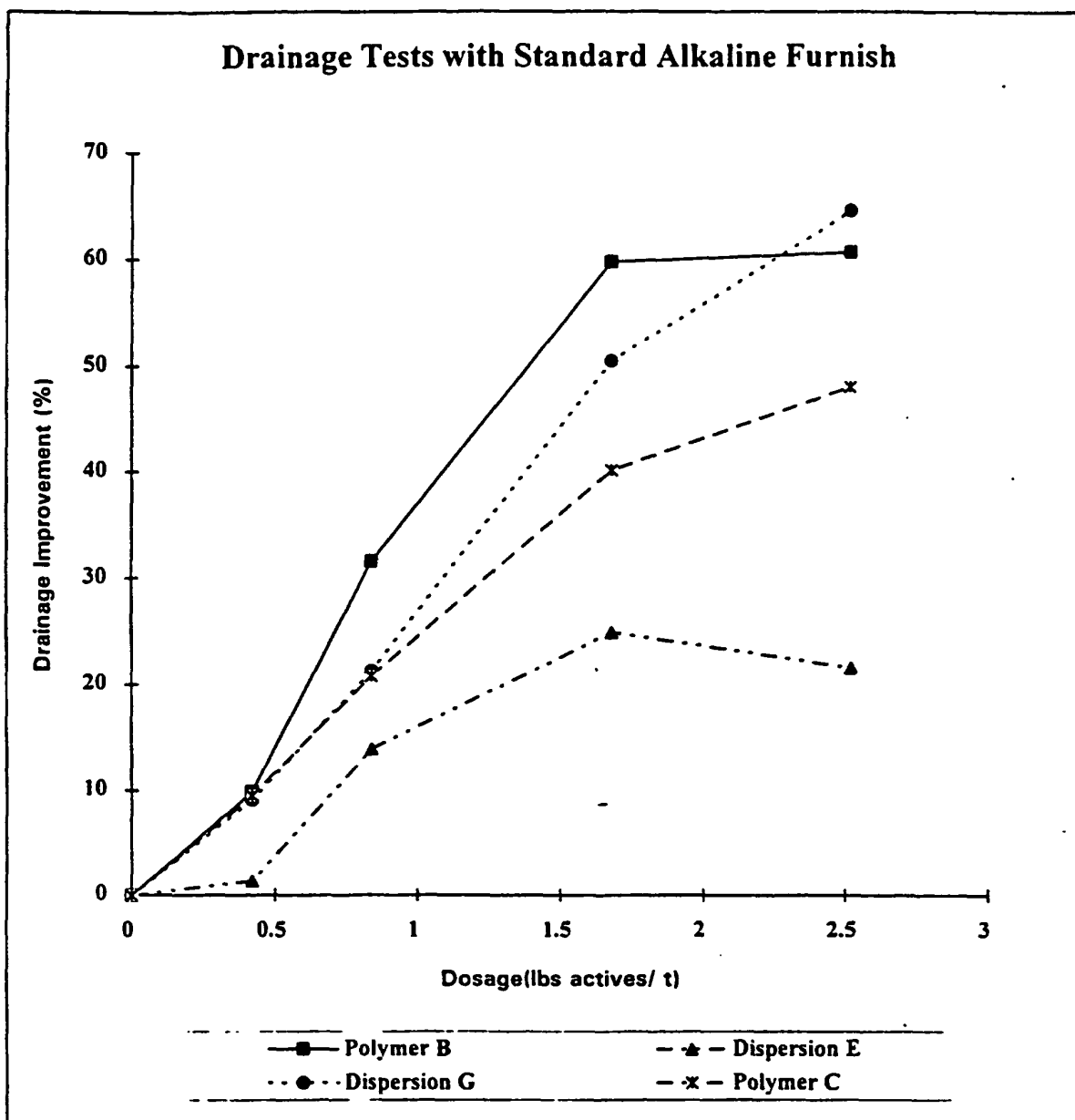
Polymer Description	Cationic Mole %	DMAEA. MCQ Mole %	DMAEA BCQ Mole %	IV 0.125N NO3 (dl/g)
Polymer A	10	10	0	17.7
Dispersion E	10	10	0	18.2
Dispersion H	10	10	0	19.2
Polymer D	70/30 mole% AcAm/NaAc			-

FIGURE 8



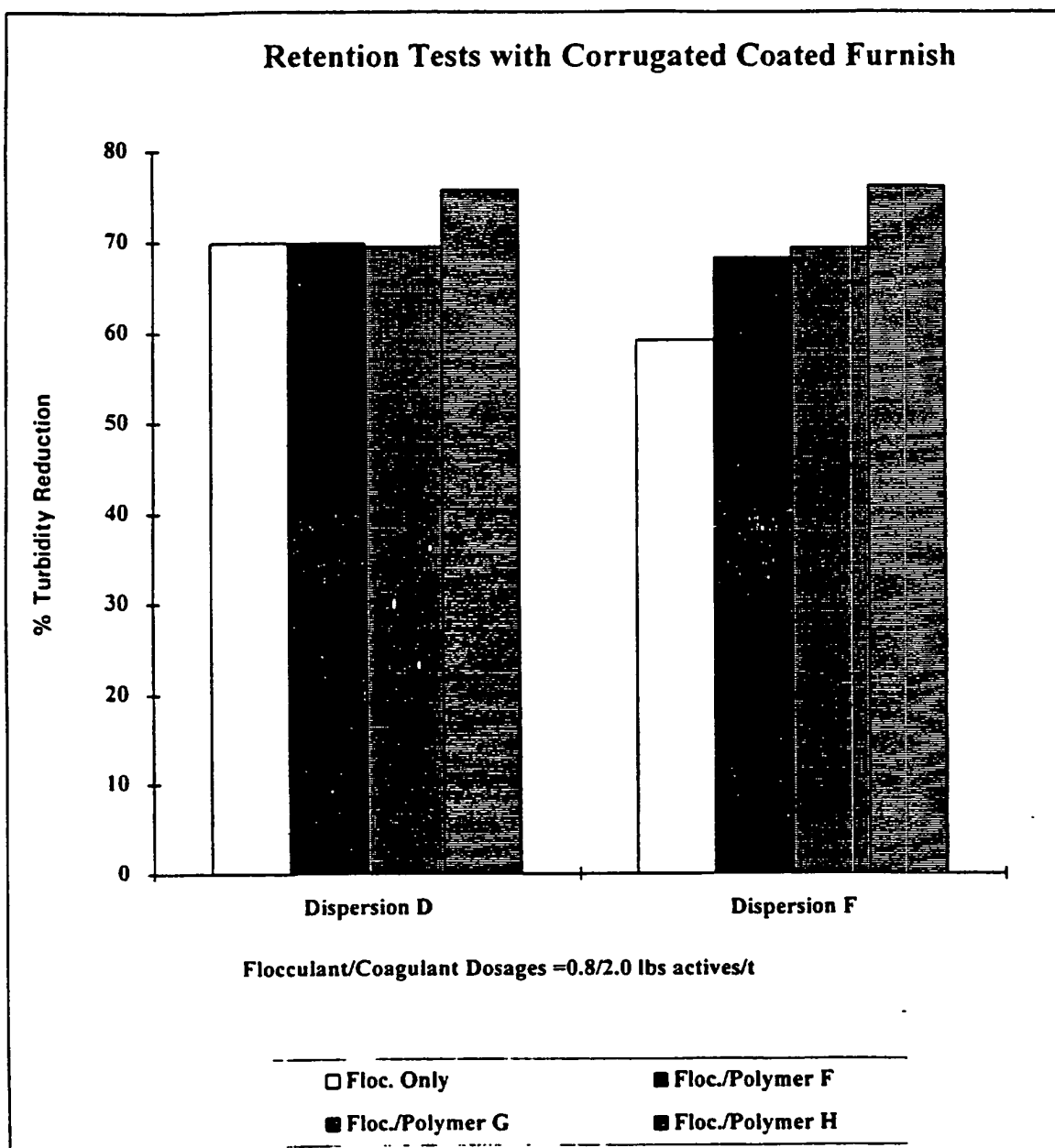
Polymer Description	Cationic Mole %	DMAEA. MCQ Mole %	DMAEA BCQ Mole %	IV 0.125N NO3 (dl/g)
Polymer B	10	10	0	19.1
Dispersion E	10	10	0	18.2
Dispersion G	20	20	0	19.4
Polymer C	10	0	10	12.9

FIGURE 9



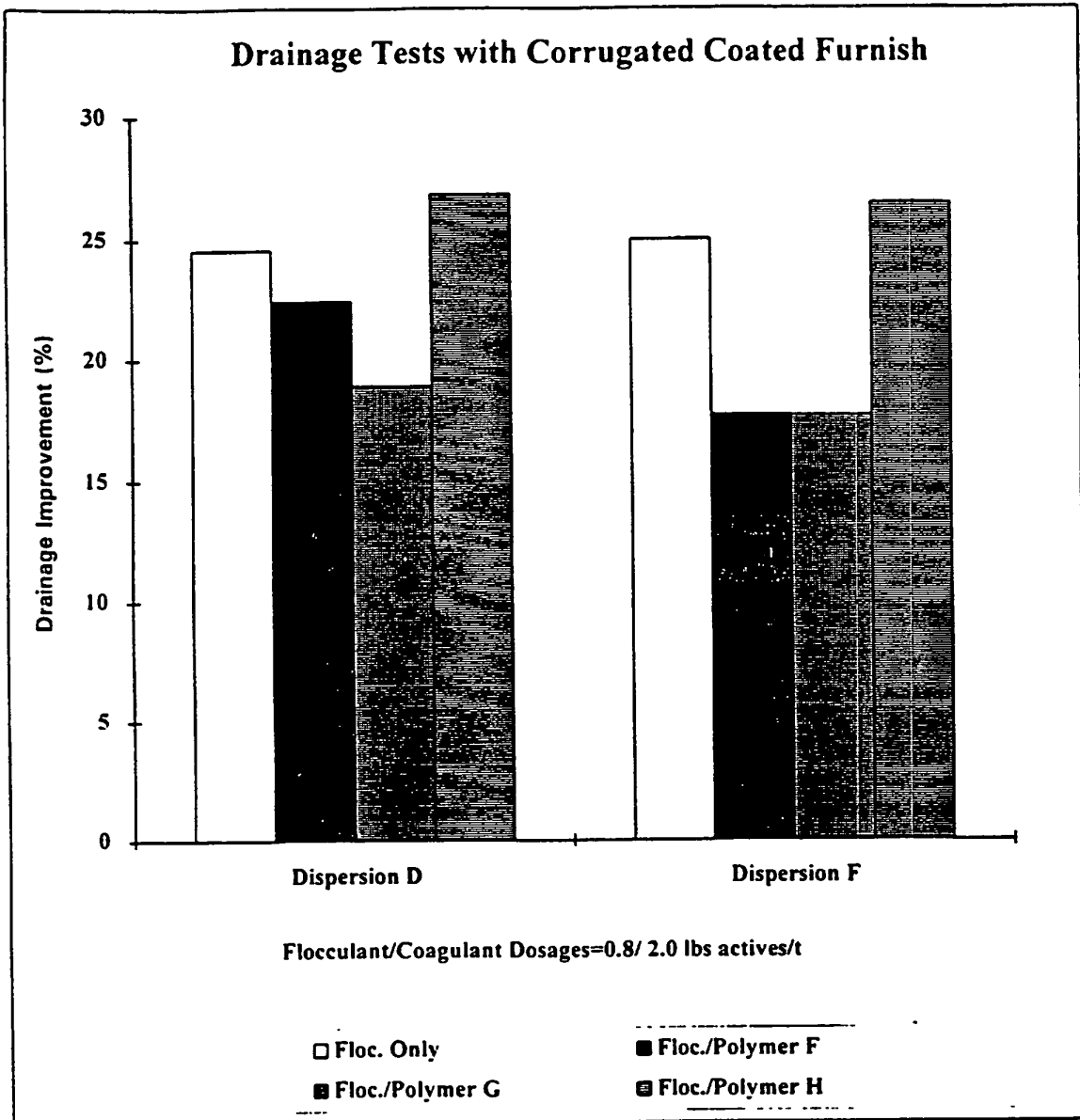
Polymer Description	Cationic Mole %	DMAEA. MCQ Mole %	DMAEA BCQ Mole %	IV 0.125N NO3 (dl/g)
Polymer B	10	10	0	19.1
Dispersion E	10	10	0	18.2
Dispersion G	20	20	0	19.4
Polymer C	10	0	10	12.9

FIGURE 10



Polymer Description	Type	Polymer Composition	DMAEA. MCQ Mole %	IV 0.125N NO3 (dl/g)
Dispersion D	Flocculant	AcAm/MCQ	10	17.0
Dispersion F	Flocculant	AcAm/MCQ	20	21.2
Polymer F	Coagulant	Epi/DMA	-	-
Polymer G	Coagulant	Poly(DADMAC)	-	Low
Polymer H	Coagulant	Poly(DADMAC)	-	High

FIGURE 11



Polymer Description	Type	Polymer Composition	DMAEA. MCQ Mole %	IV 0.125N NO3 (dl/g)
Dispersion D	Flocculant	AcAm/MCQ	10	17.0
Dispersion F	Flocculant	AcAm/MCQ	20	21.2
Polymer F	Coagulant	Epi/DMA	-	-
Polymer G	Coagulant	Poly(DADMAC)	-	Low
Polymer H	Coagulant	Poly(DADMAC)	-	High

REFERENCES CITED IN THE DESCRIPTION

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