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Chaloupli et al.

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(54) **BIOBASED ADDITIVE FOR
THERMOPLASTIC POLYESTERS**

(2013.01); C08K 5/14 (2013.01); C08K
5/34924 (2013.01); B29B 2009/125 (2013.01)

(71) Applicant: Queen's University of Kingston

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Kingston (CA)

CPC .. C08L 67/04; B29B 9/02; B29B 9/12; B33Y

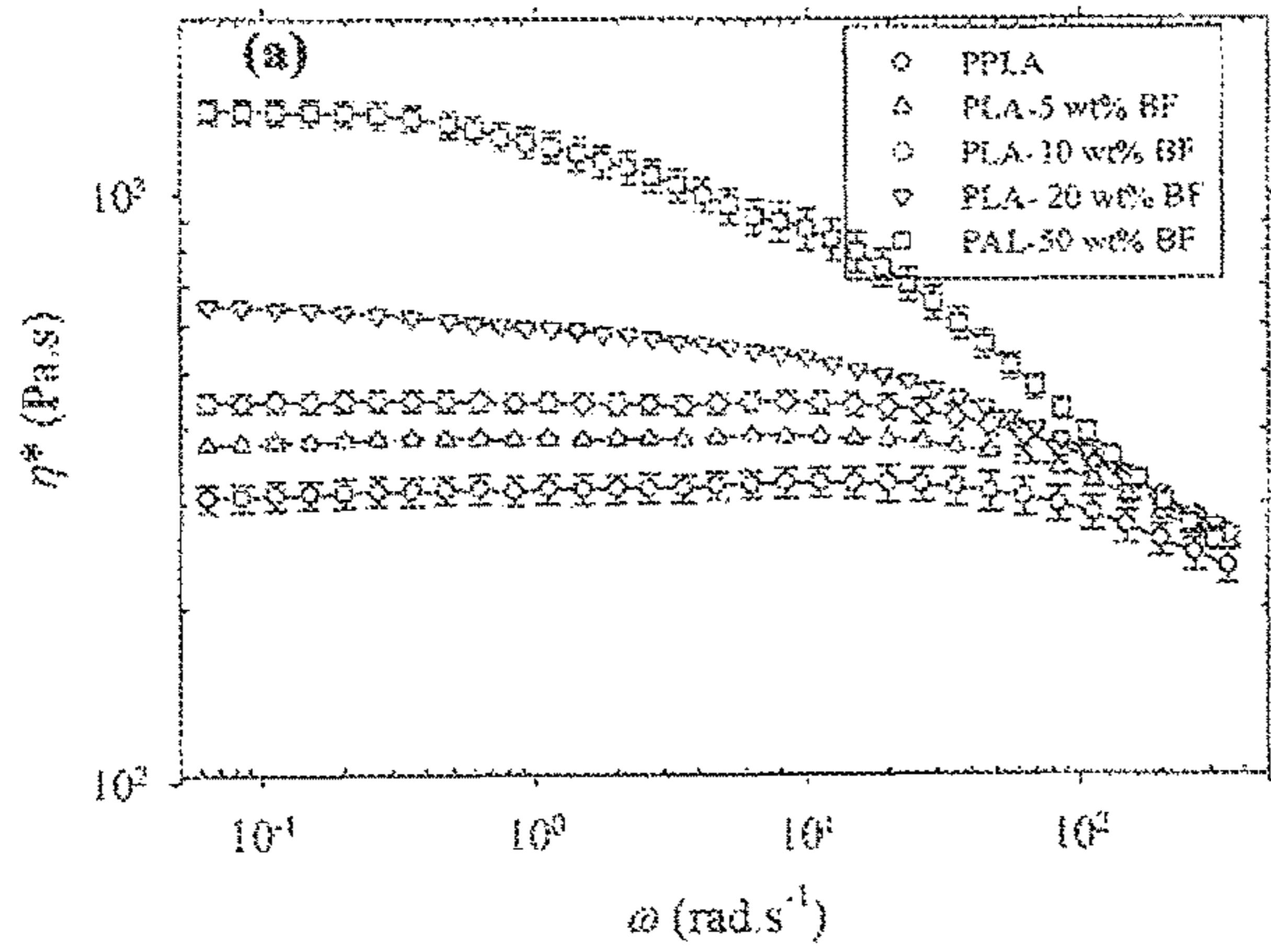


Fig. 1A

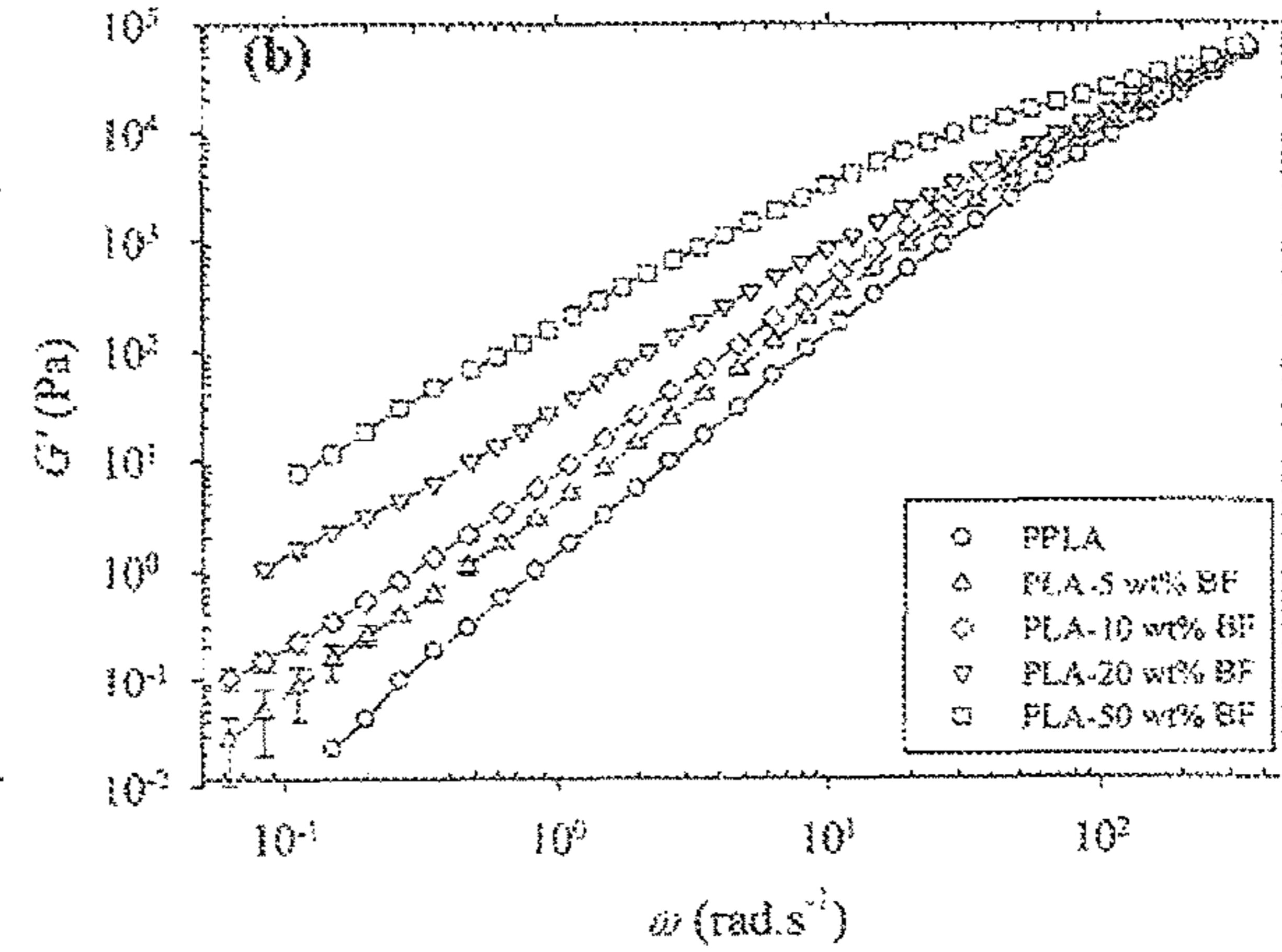
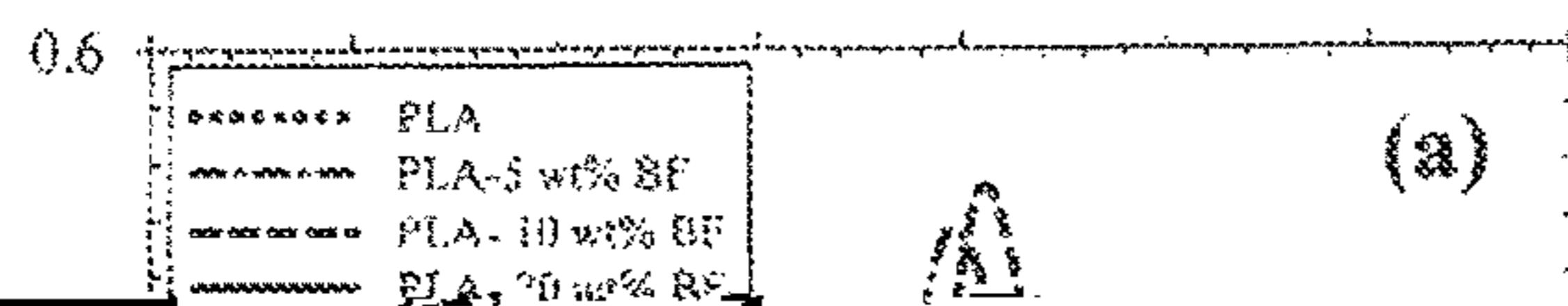
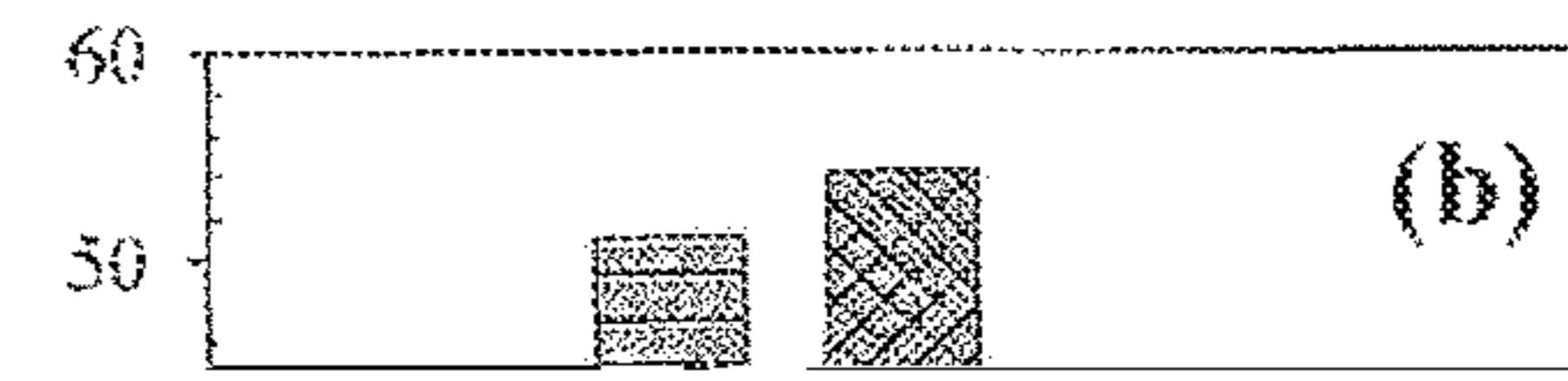


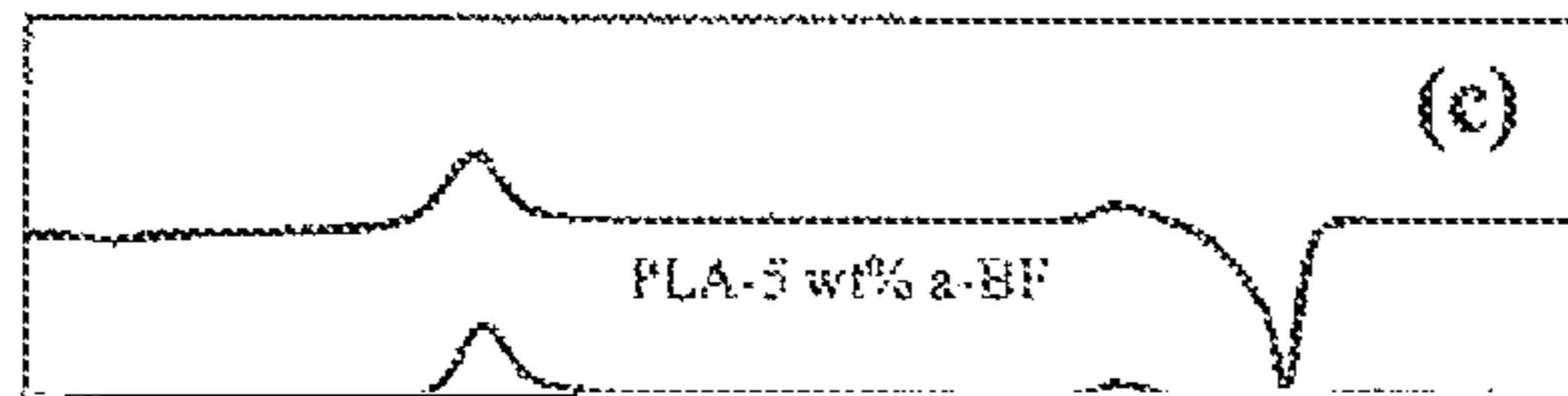
Fig. 1B

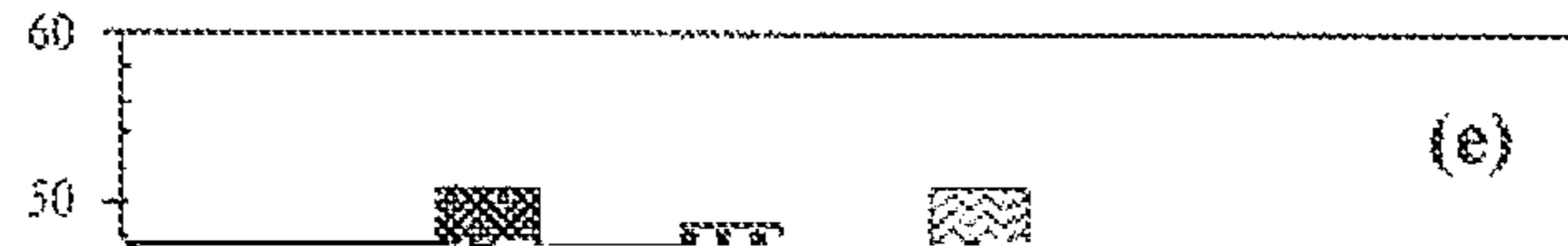


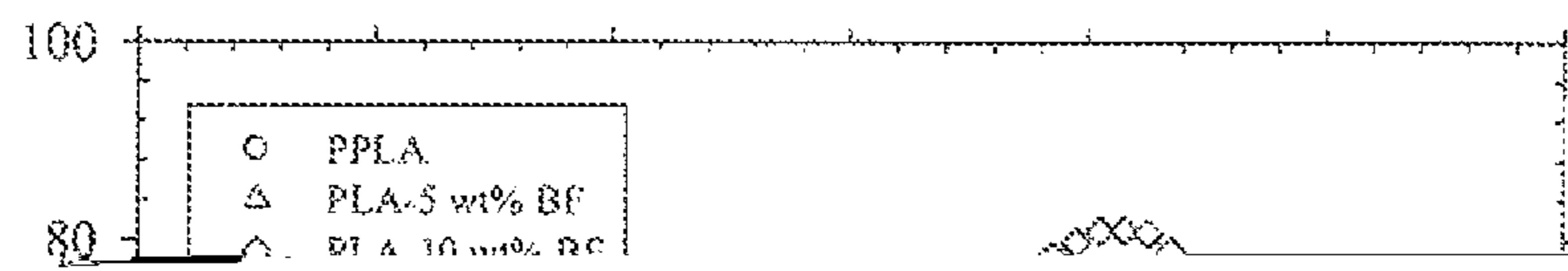
(a)

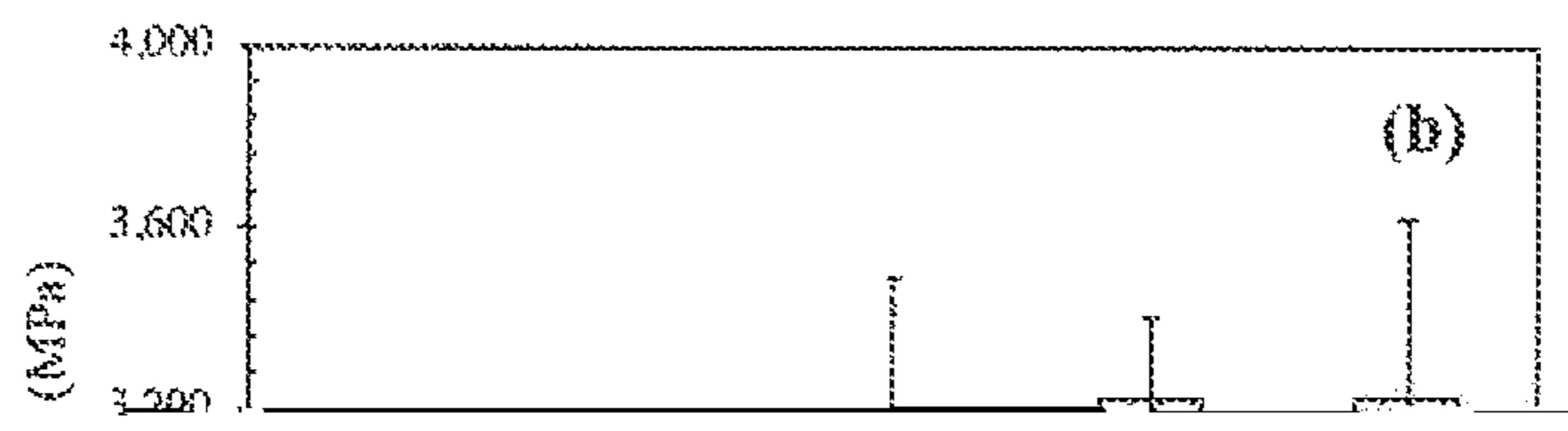


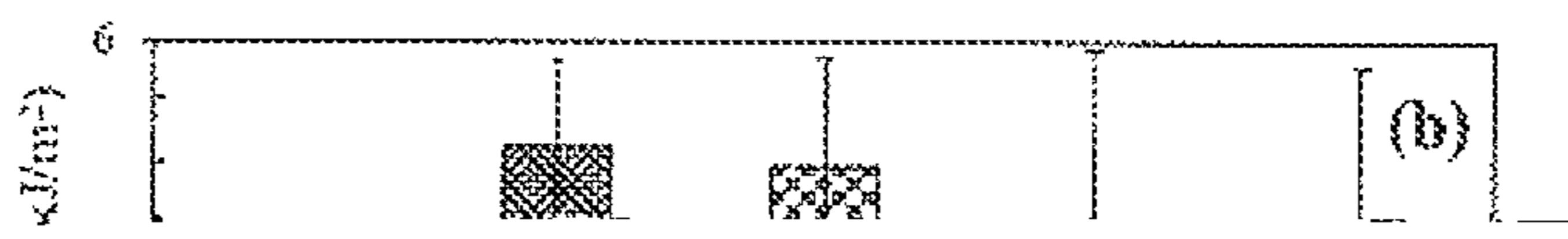
(b)












(b)



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**BIOBASED ADDITIVE FOR
THERMOPLASTIC POLYESTERS**

FIELD

The invention relates to additives for polyesters used to make thermoformed injection molded or extruded prod-

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comprising a thermoset biopolyester, wherein the particulate additive reinforces and nucleates the thermoplastic polyester. In one embodiment, the thermoplastic polyester is biodegradable or bioderived or both. In one embodiment, the thermoplastic polyester is a non-bioderived polyester. In one embodiment, the thermoplastic polyester is polyethylene

99:1, 95:5, 90:10, 80:20, 70:30, 60:40, or 50:50 by weight

In one aspect a method is provided of making the

In one embodiment, the mixing is performed in an internal batch mixer. In one embodiment, the grinding is performed

biobased additive of the above aspect, comprising heating biopolyester to a temperature sufficient to (i) melt the biopolyester and (ii) decompose a selected free radical

embodiment, the method further includes isothermal conditioning between about 80 to about 120° C.

5 initiator, adding 0.5 to 2.5 wt % of the free radical initiator and 0.5 to 2.5 wt % of a crosslinking agent to the biopoly-

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1. \rightarrow 0.4, 1.0, 1.6, 2.2, 2.8, 3.4, 4.0, 4.6, 5.2, 5.8, 6.4, 7.0, 7.6, 8.2, 8.8, 9.4, 10.0, 10.6, 11.2, 11.8, 12.4, 13.0, 13.6, 14.2, 14.8, 15.4, 16.0, 16.6, 17.2, 17.8, 18.4, 19.0, 19.6, 20.2, 20.8, 21.4, 22.0, 22.6, 23.2, 23.8, 24.4, 25.0, 25.6, 26.2, 26.8, 27.4, 28.0, 28.6, 29.2, 29.8, 30.4, 31.0, 31.6, 32.2, 32.8, 33.4, 34.0, 34.6, 35.2, 35.8, 36.4, 37.0, 37.6, 38.2, 38.8, 39.4, 40.0, 40.6, 41.2, 41.8, 42.4, 43.0, 43.6, 44.2, 44.8, 45.4, 46.0, 46.6, 47.2, 47.8, 48.4, 49.0, 49.6, 50.2, 50.8, 51.4, 52.0, 52.6, 53.2, 53.8, 54.4, 55.0, 55.6, 56.2, 56.8, 57.4, 58.0, 58.6, 59.2, 59.8, 60.4, 61.0, 61.6, 62.2, 62.8, 63.4, 64.0, 64.6, 65.2, 65.8, 66.4, 67.0, 67.6, 68.2, 68.8, 69.4, 70.0, 70.6, 71.2, 71.8, 72.4, 73.0, 73.6, 74.2, 74.8, 75.4, 76.0, 76.6, 77.2, 77.8, 78.4, 79.0, 79.6, 80.2, 80.8, 81.4, 82.0, 82.6, 83.2, 83.8, 84.4, 85.0, 85.6, 86.2, 86.8, 87.4, 88.0, 88.6, 89.2, 89.8, 90.4, 91.0, 91.6, 92.2, 92.8, 93.4, 94.0, 94.6, 95.2, 95.8, 96.4, 97.0, 97.6, 98.2, 98.8, 99.4, 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700.6, 701.2, 701.8, 702.4, 703.0, 703.6, 704.2, 704.8, 705.4, 706.0, 706.6, 707.2, 707.8, 708.4, 709.0, 709.6, 710.2, 710.8, 711.4, 712.0, 712.6, 713.2, 713.8, 714.4, 715.0, 715.6, 716.2, 716.8, 717.4, 718.0, 718.6, 719.2, 719.8, 720.4, 721.0, 721.6, 722.2, 722.8, 723.4, 724.0, 724.6, 725.2, 725.8, 726.4, 727.0, 727.6, 728.2, 728.8, 729.4, 730.0, 730.6, 731.2, 731.8, 732.4, 733.0, 733.6, 734.2, 734.8, 735.4, 736.0, 736.6, 737.2, 737.8, 738.4, 739.0, 739.6, 740.2, 740.8, 741.4, 742.0, 742.6, 743.2, 743.8, 744.4, 745.0, 745.6, 746.2, 746.8, 747.4, 748.0, 748.6, 749.2, 749.8, 750.4, 751.0, 751.6, 752.2, 752.8, 753.4, 754.0, 754.6, 755.2, 755.8, 756.4, 757.0, 757.6, 758.2, 758.8, 759.4, 760.0, 760.6, 761.2, 761.8, 762.4, 763.0, 763.6, 764.2, 764.8, 765.4, 766.0, 766.6, 767.2, 767.8, 768.4, 769.0, 769.6, 770.2, 770.8, 771.4, 772.0, 772.6, 773.2, 773.8, 774.4, 775.0, 775.6, 776.2, 776.8, 777.4, 778.0, 778.6, 779.2, 779.8, 780.4, 781.0, 781.6, 782.2, 782.8, 783.4, 784.0, 784.6, 785.2, 785.8, 786.4, 787.0, 787.6, 788.2, 788.8, 789.4, 790.0, 790.6, 791.2, 791.8, 792.4, 793.0, 793.6, 794.2, 794.8, 795.4, 796.0, 796.6, 797.2, 797.8, 798.4, 799.0, 799.6, 800.2, 800.8, 801.4, 802.0, 802.6, 803.2, 803.8, 804.4, 805.0, 805.6, 806.2, 806.8, 807.4, 808.0, 808.6, 809.2, 809.8, 810.4, 811.0, 811.6, 812.2, 812.8, 813.4, 814.0, 814.6, 815.2, 815.8, 816.4, 817.0, 817.6, 818.2, 818.8, 819.4, 820.0, 820.6, 821.2, 821.8, 822.4, 823.0, 823.6, 824.2, 824.8, 825.4, 826.0, 826.6, 827.2, 827.8, 828.4, 829.0, 829.6, 830.2, 830.8, 831.4, 832.0, 832.6, 833.2, 833.8, 834.4, 835.0, 835.6, 836.2, 836.8, 837.4, 838.0, 838.6, 839.2, 839.8, 840.4, 841.0, 841.6, 842.2, 842.8, 843.4, 844.0, 844.6, 845.2, 845.8, 846.4, 847.0, 847.6, 848.2, 848.8, 849.4, 850.0, 850.6, 851.2, 851.8, 852.4, 853.0, 853.6, 854.2, 854.8, 855.4, 856.0, 856.6, 857.2, 857.8, 858.4, 859.0, 859.6, 860.2, 860.8, 861.4, 862.0, 862.6, 863.2, 863.8, 864.4, 865.0, 865.6, 866.2, 866.8, 867.4, 868.0, 868.6, 869.2, 869.8, 870.4, 871.0, 871.6, 872.2, 872.8, 873.4, 874.0, 874.6, 875.2, 875.8, 876.4, 877.0, 877.6, 878.2, 878.8, 879.4, 880.0, 880.6, 881.2, 881.8, 882.4, 883.0, 883.6, 884.2, 884.8, 885.4, 886.0, 886.6, 887.2, 887.8, 888.4, 889.0, 889.6, 890.2, 890.8, 891.4, 892.0, 892.6, 893.2, 893.8, 894.4, 895.0, 895.6, 896.2, 896.8, 897.4, 898.0, 898.6, 899.2, 899.8, 900.4, 901.0, 901.6, 902.2, 902.8, 903.4, 904.0, 904.6, 905.2, 905.8, 906.4, 907.0, 907.6, 908.2, 908.8, 909.4, 910.0, 910.6, 911.2, 911.8, 912.4, 913.0, 913.6, 914.2, 914.8, 915.4, 916.0, 916.6, 917.2, 917.8, 918.4, 919.0, 919.6, 920.2, 920.8, 921.4, 922.0, 922.6, 923.2, 923.8, 924.4, 925.0, 925.6, 926.2, 926.8, 927.4, 928.0, 928.6, 929.2, 929.8, 930.4, 931.0, 931.6, 932.2, 932.8, 933.4, 934.0, 934.6, 935.2, 935.8, 936.4, 937.0, 937.6, 938.2, 938.8, 939.4, 940.0, 940.6, 941.2, 941.8, 942.4, 943.0, 943.6, 944.2, 944.8, 945.4, 946.0, 946.6, 947.2, 947.8, 948.4, 949.0, 949.6, 950.2, 950.8, 951.4, 952.0, 952.6, 953.2, 953.8, 954.4, 955.0, 955.6, 956.2, 956.8, 957.4, 958.0, 958.6, 959.2, 959.8, 960.4, 961.0, 961.6, 962.2, 962.8, 963.4, 964.0, 964.6, 965.2, 965.8, 966.4, 967.0, 967.6, 968.2, 968.8, 969.4, 970.0, 970.6, 971.2, 971.8, 972.4, 973.0, 973.6, 974.2, 974.8, 975.4, 976.0, 976.6, 977.2, 977.8, 978.4, 979.0, 979.6, 980.2, 980.8, 981.4, 982.0, 982.6, 983.2, 983.8, 984.4, 985.0, 985.6, 986.2, 986.8, 987.4, 988.0, 988.6, 989.2, 989.8, 990.4, 991.0, 991.6, 992.2, 992.8, 993.4, 994.0, 994.6, 995.2, 995.8, 996.4, 997.0, 997.6, 998.2, 998.8, 999.4, 1000.0, 1000.6, 1001.2, 1001.8, 1002.4, 1003.0, 1003.6, 1004.2, 1004.8, 1005.4, 1006.0, 1006.6, 1007.2, 1007.8, 1008.4, 1009.0, 1009.6, 1010.2, 1010.8, 1011.4, 1012.0, 1012.6, 1013.2, 1013.8, 1014.4, 1015.0, 1015.6, 1016.2, 1016.8, 1017.4, 1018.0, 1018.6, 1019.2, 1019.8, 1020.4, 1021.0, 1021.6, 1022.2, 1022.8, 1023.4, 1024.0, 1024.6, 1025.2, 1025.8, 1026.4, 1027.0, 1027.6, 1028.2, 1028.8, 1029.4, 1030.0, 1030.6, 1031.2, 1031.8, 1032.4, 1033.0, 1033.6, 1034.2, 1034.8, 1035.4, 1036.0, 1036.6, 1037.2, 1037.8, 1038.4, 1039.0, 1039.6, 1040.2, 1040.8, 1041.4, 1042.0, 1042.6, 1043.2, 1043.8, 1044.4, 1045.0, 1045.6, 1046.2, 1046.8, 1047.4, 1048.0, 1048.6, 1049.2, 1049.8, 1050.4, 1051.0, 1051.6, 1052.2, 1052.8, 1053.4, 1054.0, 1054.6, 1055.2, 1055.8, 1056.4, 1057.0, 1057.6, 1058.2, 1058.8, 1059.4, 1060.0, 1060.6, 1061.2, 1061.8, 1062.4, 1063.0, 1063.6, 1064.2, 10

FIG. 6. $\epsilon_{\text{eff}} = 1 + \frac{1}{11} - \frac{1}{11} \cdot \frac{1}{11} + \frac{1}{11} \cdot \frac{1}{11} - \frac{1}{11} + \frac{1}{11} - \frac{1}{11}$.

and the free-radical initiator is DCP. In one embodiment, the amount of TAM is about 1 wt % and the amount of DCP is about 1 wt %. In one embodiment, the amount of bisphenol

PLA and BF-reinforced PLA composites with specified BF loadings.

FIG. 7A graphically shows the flow and behavior of DT A and

specified in the term, for example “BF-reinforced PLA composite”.

As used herein, the term “thermoplastic”, or “thermosoftening plastic”, is a plastic material (e.g., polymer) that

poly(butylene adipate-co-terephthalate) (PBAT) and biodegradable polyesters (e.g., PCL, PBAT, PES, PBS)), the improved properties are particularly advantageous for bio-derived polyesters. Examples of thermoplastic bioderived

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loadings at a heating rate of 5° C./min. Both unconditioned

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BF-reinforced PLA composites at specified BF loadings. All

thermal properties.

Referring to FIG. 2D, a non-isothermal melt crystallization (first heating) thermograph obtained at a heating rate of 5° C./min is shown of PLA and BF-reinforced PLA composites with various BF loadings that were conditioned at

compared to PLA alone.

Referring to FIG. 9A, a graph is shown of the particle size distribution in one embodiment. Particle size is shown to range from 20 μm to 100 μm .

Referring to FIG. 9B, an optical microscopic image is

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1100-G was with concentrations of 2.5 mg m⁻¹