US006287640B1

(12) **United States Patent**
McClain et al.

(10) **Patent No.: US 6,287,640 B1**
(45) **Date of Patent: *Sep. 11, 2001**

(54) **SURFACE TREATMENT OF SUBSTRATES WITH COMPOUNDS THAT BIND THERETO**

(75) Inventors: **James B. McClain**, Raleigh; **James P. DeYoung**, Durham; **Timothy J. Romack**, Greenville, all of NC (US)

(73) Assignee: **MiCell Technologies, Inc.**, Raleigh, NC (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

This patent is subject to a terminal disclaimer.

(21) Appl. No.: **09/561,413**

(22) Filed: **Apr. 28, 2000**

Related U.S. Application Data

(63) Continuation-in-part of application No. 09/527,193, filed on Mar. 17, 2000, now Pat. No. 6,165,560, which is a continuation of application No. 09/479,566, filed on Jan. 7, 2000, now Pat. No. 6,187,383, which is a continuation of application No. 09/090,330, filed on May 29, 1998, now Pat. No. 6,030,663, which is a continuation-in-part of application No. 08/866,348, filed on May 30, 1997, now abandoned.

(51) **Int. Cl.**[7] **B05D 1/00**; B05D 7/14

(52) **U.S. Cl.** **427/388.1**; 427/389.9; 427/393.4; 427/394; 427/435; 427/439

(58) **Field of Search** 427/389.9, 393.4, 427/388.1, 439, 435, 394

(56) **References Cited**

U.S. PATENT DOCUMENTS

2,841,573	7/1958	Ahlbrecht et al.	260/79.3
3,282,905	11/1966	Fasick et al.	260/89.5
3,497,375	2/1970	Rundle et al.	117/5.1
3,811,933	5/1974	Uffner et al.	117/155
4,107,055	8/1978	Sukornick et al.	252/8.6
4,147,851	4/1979	Raynolds	526/245
4,539,006	9/1985	Langford	8/94.1
4,582,731	4/1986	Smith	427/421
4,734,227	3/1988	Smith	264/13
4,923,720	5/1990	Lee et al.	427/422
4,992,308	2/1991	Sunol	427/297
5,057,577	10/1991	Matsuo et al.	525/276
5,108,799	4/1992	Hoy et al.	427/422
5,149,753	9/1992	Inukai et al.	526/245
5,169,687	12/1992	Sunol	427/297
5,197,800	3/1993	Saidman et al.	366/136
5,199,956	4/1993	Schlenker et al.	8/473
5,211,342	5/1993	Hoy et al.	239/707
5,212,229	5/1993	Taylor et al.	524/556
5,250,078	10/1993	Saus et al.	8/475
5,269,815	12/1993	Schlenker et al.	8/475
5,290,602	3/1994	Argyropoulos et al.	427/421
5,298,032	3/1994	Schlenker et al.	8/475
5,308,648	5/1994	Prince et al.	427/212
5,326,823	7/1994	Rolando et al.	525/276
5,330,783	7/1994	Saidman et al.	427/8
5,340,614	8/1994	Perman et al.	427/2.24
5,350,795	9/1994	Smith et al.	524/507
5,362,519	11/1994	Argyropoulos et al.	427/385.5
5,407,132	4/1995	Messerly et al.	239/124
5,407,267	4/1995	Davis et al.	366/152
5,415,897	5/1995	Chang et al.	427/421
5,496,901	3/1996	DeSimone	526/89
5,508,060	4/1996	Perman et al.	427/2.14
5,512,058	4/1996	Gavend et al.	8/94.18
5,530,049	6/1996	Dee et al.	524/424
5,602,225	2/1997	Montagna et al.	528/25
5,676,705	10/1997	Jureller et al.	8/142
5,683,473	11/1997	Jureller et al.	8/142
5,683,977	11/1997	Jureller et al.	510/286

FOREIGN PATENT DOCUMENTS

3904514 A	8/1990	(DE) .
3906724 A	9/1990	(DE) .
3906737 A	9/1990	(DE) .
4332219 A	3/1994	(DE) .
42 39 214 A1	5/1994	(DE) .
4429470 A	3/1995	(DE) .
4333221 A	4/1995	(DE) .
4336941 A	5/1995	(DE) .
4344021 A	6/1995	(DE) .
4404839 A	8/1995	(DE) .
492535	7/1992	(EP) .
0506 067 A1	9/1992	(EP) .
WO 93/14255	7/1993	(WO) .
WO 93/14259 A	7/1993	(WO) .
WO 97/16264	5/1997	(WO) .
WO 98/11293	3/1998	(WO) .

OTHER PUBLICATIONS

Rao et al., Textile Finishes and Fluorosurfactants, *Organofluorine Chemistry: Principles and Commercial Applications,* Banks et al. (eds), Plenum Press, New York, pp. 321–338 (1994).

Bowman et al., Sizing and Desizing Polyester/Cotton Blend Yarns Using Liquid Carbon Dioxide, *Textile Res. J.,* 66 (12):795–802 (1996).

DeSimone et al., Synthesis of Fluoropolymers in Supercritical Carbon Dioxide, *Science,* 257:945–947 (1992).

AATCC's 1997 Int'l Conference & Exhibition; XP–000722163, Speaker—Joseph M. DeSimone, Surfactants for Liquid and Supercritical Carbon Dioxide, *Textile Chemist and Colorist,* 29(8):28,30 (Aug. 1997).

International Search Report for PCT/US98/10897, dated Apr. 19, 1998.

Primary Examiner—Erma Cameron
(74) *Attorney, Agent, or Firm*—Myers Bigel Sibley & Sajovec

(57) **ABSTRACT**

A method of treating a substrate comprises contacting a surface of said substrate with a pressurized fluid comprising carbon dioxide and a surface treatment component, the surface treatment component being one which binds to the substrate. The contacting step is preferably carried out by immersion, the fluid is preferably a liquid or supercritical fluid, the substrate is preferably a metal or fabric substrate, and the surface treatment component is preferably a fluoroacrylate polymer.

43 Claims, No Drawings