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Quality Control Studies on Multi-Sourced Aspirin Tablets Manufactured in Nigeria.

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ABSTRACT

Aspirin is one of the safest and least expensive over the counter drugs (OTCs) used in the treatment of head ache, general pain or fever, for the prevention of strokes and for diseases such as rheumatic fever, gout and rheumatoid arthritis. The aim of the study was to assess the *in vitro* quality control parameters of ten brands of aspirin tablets (BP 300 mg) sourced from community and hospital pharmacies in three Nigerian cities by evaluating their organoleptic and physicochemical properties such as identity test, weight variation, hardness, friability, disintegration time, dissolution rate and drug content. Results obtained revealed that all the ten brands of aspirin were identified and had uniform organoleptic properties. Two samples (C and H) failed the uniformity of weight test as the percentage deviation of their individual weights from the average was outside the BP 2005 stated range of ± 5.0 %. Three samples (B, F and I) failed the friability test with percentage losses of 2.48, 2.06, and 4.3 respectively. One sample (sample I) failed the disintegration time test as it took more than 15 mins for it to disintegrate. The percentage drug content of all the samples was within the official range of 95 – 105 %. In conclusion, routine quality control studies on all drugs circulating in Nigeria should be accertained.

KEYWORDS: Aspirin tablets, Quality control, Pains, Disintegration test, Organoleptic properties.

INTRODUCTION

Aspirin (acetyl salicylic acid) is one of the safest and least expensive over the counter (OTC) drug that is available in several dosage forms such as tablets, capsules, suppositories and elixirs. It has analgesic, antipyretic and anti-inflammatory properties and is a component of several pain relieving mixtures. Aspirin is indicated for headache, general pain or fever, for the prevention of heart attacks and strokes; and for diseases such as rheumatic fever, gout and rheumatoid arthritis [1]. Because of its availability, effectiveness, affordability, low toxicity and less side effects, it is the most commonly used of all analgesics [2]. The aim of any drug therapy is to achieve maximum benefit with minimum side effects [3]. This can only be achieved with the availability of competent healthcare professionals and good quality drugs. To ensure that drugs conform to the right standard of purity, efficacy and safety, drug manufacturers are obliged to follow Good Manufacturing Practices (GMP) and current Good Manufacturing practices (cGMP). Furthermore, relevant government agencies such as NAFDAC (National Agency for Food, Drug Administration and Control), the Nigerian equivalent of the Food and Drug Administration in USA, carryout routine quality control tests on both locally manufactured and imported pharmaceuticals to ensure that drugs circulating in Nigeria meet official standards. In most third world countries, the market for counterfeit and substandard pharmaceuticals is lucrative because of non existence of a reliable drug distribution system and the inability of the governments of such countries to enforce drug laws. In addition, the lack of good manufacturing practice (GMP) is common in local pharmaceutical industries in the Third world countries because of many hurdles such as epileptic power supply and shortage of water [4]. The stability of drugs marketed in the tropics with generally poor storage conditions, high temperature and high humidity enhances chemical degradation that may alter the biopharmaceutical properties of the drugs [5, 6]. Although drug counterfeiting is a global problem, it is rife in the Third world countries

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where drug regulations are weak or nonexistent. Following the death of 109 children in the 90s from fake paracetamol [7], the Nigerian government took steps towards prohibiting the sale and distribution of fake and substandard drugs in the country. It also established the National Agency for Food, Drug Administration and Control (NAFDAC) in 1993 to among other functions regulate and control the importation, exportation, manufacture, advertisement, distribution, sale and use of food, drugs and pharmaceutical products. The agency also conducts appropriate tests to ensure compliance with official specifications for effective control of drug quality [8]. There is therefore a need to routinely assess the pharmaceutical quality of drugs available in Nigeria. In this study, the quality control parameters studied were organoleptic characteristics, weight uniformity, hardness/crushing strength, friability, disintegration, dissolution and percentage content of ten brands of aspirin tablets using official and unofficial methods.

MATERIALS AND METHODS

Chemicals and drugs

Ten brands of aspirin tablets (BP 300 mg) were purchased from various patent medicine shops in Owerri, Elele and Port Harcourt. The drugs were coded A – J and information such as brand name, batch number, labeled strength, manufacturing and expiry dates, NAFDAC registration status were noted (Table 1). All other chemicals used were of analytical grade. Distilled water was obtained from our laboratory and used as such.

Organoleptic properties

The following organoleptic properties were assessed by three independent persons: shape, colour, taste and smell.

Physicochemical properties

Identity tests

Aspirin in the tablets was identified using the British Pharmacopoeia method [9]. 0.5 g of the powdered tablets of each brand was boiled with 10 ml of 5 M NaOH for 3 min, cooled and 2 ml of 1 M H_2SO_4 added. A crystalline precipitate was produced. To a 5 ml solution of the precipitate in water, 1 ml of $FeCl_3$ solution was added and observed for a deep violet coloration.

Weight variation

Twenty tablets of each brand were weighed individually with an electronic balance (Mettler Toledo, Switzerland) and the mean and standard deviation calculated. The percentage deviation was also determined.

Hardness / Crushing strength

A hardness tester (Vickers Ltd, London) was used on ten tablets of each brand and the mean crushing strength was calculated.

Friability

Ten tablets from each brand were weighed and put in a B and T Tablet friability tester (Erweka TAR Germany) and rotated at 25 rpm. The friability (percentage) was calculated for each brand.

Disintegration time

The disintegration time of the tablets were determined in distilled water at 37 ± 0.5^{o}C using an Avis single unit disintegration test apparatus (G.B. Caleva Ltd, model 917, England)

Dissolution rate

An Avis single unit dissolution test apparatus (G.B. Caleva Ltd, model 917, England) was used and the method employed was the paddle method as outlined in the British Pharmacopoeia, [9]. The stirring rate was 75 rpm with 900 ml of distilled water as the medium. The samples were withdrawn at definite time intervals for 1 h and assayed spectrophotometrically at 265 nm.

Assay of tablets

The BP 2005 method was used. Twenty tablets of each brand of aspirin was weighed and to the quantity of the powder containing 0.5 g of the drug was added 30 ml of 0.5 M NaOH and boiled under reflux for 10 min. It was back titrated with 0.5 M HCl using phenol red as indicator. A blank determination was carried out. The amount of aspirin was calculated and expressed as a percentage.

RESULTS

The ten brands were positively identified by the production of violet coloration on hydrolysis with NaOH and the subsequent addition of ferric chloride. Results of the organoleptic evaluation of the ten brands of aspirin tablets involved in the study are summarized in Table 2. The ten brands of aspirin complied with the official standards of organoleptic properties. They were all odorless, spherical, white in color and bitter. Results of the assessment of physicochemical properties showed that two samples C and H failed the weight uniformity test as the percentage deviations of the individual weights from the average was outside the BP 2005 stated range of ± 5.0 %.

Table 1: Label information on the tablets involved in the study

Sample Code	Trade name	Months to Expiration	NAFDAC Reg. status	Place of purchase	Source of Drugs	Place of manufacture
A	Anacin	14	Positive	Asaba	CP	Lagos
B	Dispirin	13	Positive	Owerri	HP	Lagos
C	Propon	13	Positive	Asaba	CP	Lagos
D	Aspirin	14	Positive	Owerri	CP	Lagos
E	Aspirin	12	Positive	Owerri	HP	Lagos
F	Aspirin	14	Positive	Aba	CP	Lagos
G	Aspirin	14	Positive	Aba	HP	Enugu
H	Aspirin	12	Positive	Asaba	CP	Lagos
I	Aspirin	10	Positive	Aba	HP	Lagos
J	Aspirin	11	Positive	Owerri	CP	Lagos

CP = community pharmacy HP = Hospital pharmacy

Table 2: Organoleptic properties of aspirin tablets

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Sample code	Shape	Colour	Taste	Smell
A	Round	White	Bitter	Odourless
B	Round	White	Bitter	Odourless
C	Round	White	Bitter	Odourless
D	Round	White	Bitter	Odourless
E	Round	White	Bitter	Odourless
F	Round	White	Bitter	Odourless
G	Round	White	Bitter	Odourless
H	Round	White	Bitter	Odourless
I	Round	White	Bitter	Odourless
J	Round	White	Bitter	Odourless

Table 3: Physicochemical properties of aspirin tablets involved in the study

Brand code	I_D(colour)	Wt (mg)	H_T(Kg/f)	F_T(%)	F_T(%)	D_S(%)	Assay(%)
A	+ve	0.457	3.51	0.65	4	84	101
B	+ve	0.444	5.74	2.48	7	77	96
C	+ve	0.630	5.19	0.70	12	78	96
D	+ve	0.368	6.86	0.70	6	81	102
E	+ve	0.370	4.87	0.20	14	76	101
F	+ve	0.330	5.49	2.06	4	95	102
G	+ve	0.343	4.03	0.70	11	77	99
H	+ve	0.574	4.77	0.20	6	85	103
I	+ve	0.327	6.55	4.30	19	-	95
J	+ve	0.335	6.50	0.60	12	89	99

I_D= Identification test W_t= weight uniformity H_T = Hardness / crushing strength F_T = Friability
F_T = Friability D_T = Disintegration time D_S = Dissolution rate (%) within 45 mins.

The crushing strengths of the aspirin tablets analyzed ranged from 3.5 to 7.0, with sample A having the least crushing strength and sample I having the highest. Although not an official requirement the crushing strength must be controlled to ensure that the product is firm enough to withstand handling without breaking or crumbling and not so hard that the disintegration time is unduly prolonged [10]. Three samples B, F and I failed the friability test with percentage losses of 2.48, 2.06 and 4.30 respectively. The British Pharmacopoeia stipulates that the percentage loss should be less than or equal to 1[9].

The disintegration times for all the aspirin tablets ranged from 4 to 19 min. One sample, I, failed the disintegration test as it took more than 15 min to disintegrate. The percentage dissolution of all the samples of the aspirin tablets in the study was within the pharmacopoeia range of at least 70 % of stated amount within 45 min. The content of aspirin in all the samples analysed was within the official range of 95 – 105 %.

DISCUSSION

Nigeria has a large market for drugs and it also serves as a hub for drug distribution in the West African sub region, highlighting the need for all pharmaceuticals in Nigeria to comply with official specifications for drug quality. *In vitro* laboratory testing together with *in vivo* studies are both used to ensure the compliance of manufacturers with official specifications for quality. One tablet failed the disintegration time test as it took more than 15 min to disintegrate. The BP 2005 specifies a disintegration time of not more than 15 min for uncoated tablets [9]. Disintegration measures the time required for the tablets to disintegrate into particles prior to dissolution [11]. The poor disintegration of sample I could have resulted from the excessive use of binders and inadequate use of disintegrants during tablet manufacture, hence the tablet core would be strongly held together preventing penetration of disintegration fluid [12]. Dissolution measures the release characteristics of active ingredients *in vitro* from solid dosage forms and it gives a fair indication of bioavailabilty [11]. All the samples involved in the dissolution test met the BP specification which states that at 45 min all tablets and capsules should release at least 70 % of the total amount of active ingredient into the dissolution medium. Sample I failed the disintegration time test so it was not included in the dissolution studies. One brand failed the uniformity of content. The reason could be due to poor in-process control during manufacturing as well as inadequate weighing and mixing during preparation [12].

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