

## Improving the bioactive functionality of barley dietary fibre by chemical treatments in combination with extrusion process

\*Bader Ul Ain, H., Saeed, F., Arshad, M.U., Ahmad, N., Niaz, B. and Tufail, T.

*Institute of Home and Food Sciences, Government College University Faisalabad, Kotwali Rd, Gurunanakpura, Faisalabad, Punjab 38000, Pakistan.*

### Article history

Received: 10 January, 2018

Received in revised form:

12 July, 2018

Accepted: 11 August, 2018

### Abstract

The present work was carried out to observe the comparative effectiveness of different chemical treatments in combination with simultaneous thermal treatment on soluble and insoluble dietary fibre ratio to improve functional properties of barley. For this purpose, two varieties of barley i.e., Haider-93 and Jau-87 were assessed for their chemical composition, mineral, non-starch polysaccharides and dietary fibre contents through respective methods. Next, both varieties were chemically treated with acid, alkaline and consecutive acid-alkaline treatments in combination with thermal treatment. Results of chemical composition revealed that Jau-87 was higher in moisture (11.4%), crude fat (2.67%) and crude fibre (4.70%) whereas Haider-93 exhibited higher ash (2.56%) and crude protein content (12.7%). Moreover, barley was rich source of potassium ranging from 4.77-5.07 g/kg. Likewise, main non-starch polysaccharides in barley were arabinoxylan (3.60-3.77%) and  $\beta$ -glucan (3.65-3.67%). Furthermore, barley contained more insoluble dietary fibre (12.00-12.40 g/100 g dm) than soluble dietary fibre (4.73-5.70 g/100 g dm). Additionally, modification of soluble (1.48%) and insoluble dietary fibre (8.71%) ratio through extrusion processing was non-significant whilst acid-alkaline treatment showed highly significant results i.e., 771.46% increase in soluble dietary fibre and 53.39% decrease in insoluble dietary fibre. It was therefore concluded that chemical treatments alone or in combination with twin-screw extrusion increased soluble dietary fibre. However, simultaneous effect of acid and alkaline treatment most effectively increased the solubility of barley.

### Keywords

Barley

Dietary fibre

Extrusion

Chemical modification

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## Introduction

In the past few years, efforts have been aimed to determine the scientific justification for functional properties of dietary fibre (Jha and Berrocoese, 2015). Dietary fibre refers to the non-digestible carbohydrate entity which is impervious to enzymatic assimilation and absorption in small intestine of humans. It is conventionally classified in two categories according to their water solubility: insoluble dietary fibre and soluble dietary fibre (Lattimer and Haub, 2010; Dhingra *et al.*, 2012). Each fraction has its own physiological effects (Fuller *et al.*, 2016). Soluble fractions are fermented faster into short chain fatty acids, and are consumed by the beneficial intestinal bacteria than insoluble fractions. From functional point of view, soluble dietary fibre is more important than insoluble dietary fibre. In this respect, soluble dietary fibre has been found to be significant in reducing the high cholesterol, triglyceride and glucose

levels in blood through binding. These can also affect the texture, gelling, thickening and emulsifying properties of foods (Anderson, 2009). Approximately 20-35% of soluble dietary fibres and 65-80% of insoluble dietary fibres are present in foods (mainly cereals). As far as the RDA ratio is concerned, one quarter of soluble dietary fibre and three quarters of insoluble dietary fibre (1:3) is recommended (Burton-Freeman *et al.*, 2017). The main sources of dietary fibres are cereals, nuts, fruits and vegetables (Jha and Berrocoese, 2015). Barley (*Hordeum vulgare* L.) is one of the richest sources for fibres among the cereal group, as it contains about 24% dietary fibre in the ratio of 3:2 for insoluble dietary fibre and soluble dietary fibre (Feng *et al.*, 2017).

Chemical treatment has been considered as the most important approach among all techniques because chemical reagents cause less damage to the molecular structure of the dietary fibre, and the content of soluble dietary fibre could be increased

\*Corresponding author.

Email: humahums@yahoo.com

more efficiently. Chemical methods use acid and alkali to solubilise the dietary fibre of cereals (Huang *et al.*, 2015). It depends upon the amount of acid and alkali, temperature and reaction time. Englyst and Cumming (1984) used sulphuric acid and trifluoroacetic acid to hydrolyse hemicellulose. This treatment hydrolyses the long polysaccharide chain to smaller fractions to increase their solubility. Huang and Ma (2016) applied high temperature, pressure and shear force to gasify and extend the moisture content present in the cereals (Jing and Chi, 2013; Huang and Ma, 2016). This mechanism depends upon the processing parameters such as temperature and pressure (Chen *et al.*, 2014; Rashid *et al.*, 2015). In general, a combined method might have greater effect on the modification of insoluble dietary fibre into soluble dietary fibre in cereal than the use of the single method (Ma and Mu, 2016; Tang *et al.*, 2016).

Keeping in mind all the aforementioned perspectives, there is a need to partially convert this insoluble dietary fibre into soluble dietary fibre, and to develop the soluble dietary fibre enriched value added barley products. The objective of the present work was therefore to evaluate the comparative and synergistic effect of chemical treatment and extrusion cooking on the modification of insoluble dietary fibre into soluble dietary fibre in two barley varieties.

## Materials and methods

### *Procurement of raw material*

Two barley varieties i.e., Haider-93 and Jau-87 were procured from Ayub Agriculture Research Institute (AARI), Faisalabad, Pakistan. The seeds were cleaned to remove any debris and field dirt before sealed in polyethylene bags.

### *Chemical composition*

The chemical composition of barley varieties was assessed to check the contents of moisture, ash, crude fat, crude protein, crude fibre and nitrogen free extract according to the method of AOAC (2005).

### *Mineral content*

The mineral contents such as Na, K, Ca, Mg, Fe, Cu, Zn and Mn in both barley varieties were determined by the method described in AOAC (1990).

### *Non-starch polysaccharides content*

The non-starch polysaccharides content like arabinoxylan and  $\beta$ -glucan in barley varieties was assessed by the method of Pettolino *et al.* (2012) and AOAC Method 995.16.

### *Dietary fibre content*

The contents of dietary fibre in both barley varieties were determined according to the method of AOAC 991.43 enzymatic gravimetric method (AOAC, 2005).

### *Chemical treatments*

The dietary fibre from barley varieties were chemically modified and extruded for the partial conversion of insoluble dietary fibre into soluble dietary fibre according to the method of Ning *et al.* (1991). In brief, a mixture of barley fibre and water, at a ratio of 1:5 was used. Its pH values were adjusted acidic (pH 2.0-4.0) and alkaline (pH 9.0-11.0) by using 6.0 N HCl (acid treatment) and 6.0 N NaOH (alkaline treatment), respectively. After pH adjustment, the mixtures were then heated at 90°C for different periods of time ranging from 1 to 4 h. At the end of each treatment, the supernatant was removed, neutralised, and then centrifuged at 700 g for 10 min. Pentose and hexose contents were measured for the acid treatment using high-performance liquid chromatography, and total sugar content was determined for the alkaline treatment according to the procedure of Folkes and Taylor (1982) and Dubois *et al.* (1956) respectively. The precipitate was washed with water and dried using an air drier (Proctor Schwartz, Philadelphia, PA) at 75°C for 2 h, followed by grinding and screening through a 2-mm sieve. The acid-alkaline treatment involved consecutive acid and alkaline treatments, according to the aforementioned procedures. For the acidic treatment, the mixture was adjusted to pH 2.0 with 6.0 N HCl, while for the alkaline treatment; the mixture was adjusted to pH 11.0 with 6.0 N NaOH.

### *Extrusion of unmodified and chemically modified barley fibre*

Unmodified (untreated; control) and acid- and alkaline-treated barley fibre was extruded using a ZSK-30 twin-screw extruder (Werner and Pfleiderer Crop., Ramsey, NJ) according to Ning *et al.* (1991). Conditions of extrusion were selected according to preliminary work to ensure conditions suitable for bran modification. The barrel temperatures of the first two sections were maintained at 40 and 90°C, and the remaining three sections were held at 120°C. Extrusion was carried out at 50% moisture and a dry feed rate of 200 g/min. The screw speed used was 350 rpm. A dual-orifice die (3 mm in diameter) was used. The extrudate was gently dried in an air drier at room temperature for 24 h before ground and sieved.

### Statistical analysis

The data obtained for each parameter was subjected for completely randomised design (CRD) and later on Analysis of Variance (ANOVA) to determine the level of significance (Steel *et al.*, 1997).

### Results and discussion

Chemical composition and mineral profile of barley varieties

Mean values regarding the barleys' chemical compositions are shown in Table 1. The results indicated that Jau-87 exhibited relatively high moisture (11.4%), crude fat (2.67%) and crude fibre (4.70%) contents than Haider-93 (i.e., 10.2, 2.47, 4.53% respectively). Haider-93 was higher in ash (2.56%), crude protein (12.7%) and NFE (67.55%) contents as compared to Jau-87 that contained about 2.35, 12.13, 66.64% ash, crude protein and NFE content, respectively. The results are in accordance with the earlier findings of Labouar *et al.* (2017) who reported 10.8%, 2-4%, 11.37% and 3.82% moisture, ash, crude protein and crude fat in barley, respectively. Similarly, Lee *et al.* (2016) found 10.10-13.10% moisture, 2.02-2.38% ash, 9.46-11.33% crude protein, 3.90-5.81% crude fat and 1.70-2.18% ether extract in barley, respectively.

Table 1 shows the mean values for macro- and micro-mineral contents of the two different barley varieties. In Jau-87, macro-minerals *i.e.*,

potassium, magnesium, calcium and phosphorus were 4.77, 1.31, 0.44 and 3.78 g/kg, respectively, whereas in Haider-93, 5.07, 1.35, 0.51 and 4.08 g/kg, respectively. However, micro-minerals including copper, manganese, iron and zinc were 7.93, 13.40, 49.40 and 23.50 g/kg in Jau-87, and 8.70, 14.50, 52.87 and 24.43 g/kg in Haider-93, respectively. The results are in line with the observation of Boila *et al.* (1993) who reported that barley contained calcium, phosphorus, magnesium, potassium, copper, zinc, manganese and iron in the ranges of 0.39-0.56, 3.69-4.48, 1.28-1.41, 4.70-5.31, 7.5-9.6, 22.9-32.3, 12.8-16.2 and 48.1-58.3 g/kg, respectively.

### Non-starch polysaccharides content of barley varieties

Mean values for monosaccharide content of two different varieties of barley are listed in Table 1. Arabinoxylan content (8.35%) was more in Haider-93 than in Jau-87 (8.21%), whereas  $\beta$ -glucan content was 3.67 and 3.65% in Jau-87 and Haider-93, respectively. These results are similar to the study of Andersson *et al.* (1999) who reported 75-90 and 28-69 g/kg dm of arabinoxylan and  $\beta$ -glucan in barley. Likewise, in another study, arabinoxylan and  $\beta$ -glucan content was 8.4 and 4.1%, respectively in barley (Knusen, 1997; McCleary and Glennie-Holmes, 1985).

Table 1: Mean values of (a) chemical composition, (b) mineral profile and (c) non-starch polysaccharide contents of barley varieties

| Barley varieties | (a) Chemical composition (%) |             |             |               |             |              |
|------------------|------------------------------|-------------|-------------|---------------|-------------|--------------|
|                  | Moisture                     | Ash         | Crude fat   | Crude protein | Crude fibre | NFE          |
| Jau-87           | 11.4 ± 0.61                  | 2.35 ± 0.03 | 2.67 ± 0.25 | 12.13 ± 0.65  | 4.70 ± 0.07 | 66.64 ± 0.50 |
| Haider-93        | 10.2 ± 0.3                   | 2.56 ± 0.10 | 2.47 ± 0.35 | 12.7 ± 0.8    | 4.53 ± 0.09 | 67.55 ± 1.06 |
| Mean             | 10.8                         | 2.46        | 2.57        | 12.42         | 4.62        | 67.10        |

| Barley varieties | (b) Mineral           |             |             |             |                       |              |              |              |
|------------------|-----------------------|-------------|-------------|-------------|-----------------------|--------------|--------------|--------------|
|                  | Macro-minerals (g/kg) |             |             |             | Micro-minerals (g/kg) |              |              |              |
|                  | K                     | Mg          | Ca          | P           | Cu                    | Mn           | Fe           | Zn           |
| Jau-87           | 4.77 ± 0.06           | 1.31 ± 0.05 | 0.44 ± 0.05 | 3.78 ± 0.07 | 7.93 ± 0.45           | 13.40 ± 0.56 | 49.40 ± 1.30 | 23.50 ± 0.60 |
| Haider-93        | 5.07 ± 0.06           | 1.35 ± 0.06 | 0.51 ± 0.04 | 4.08 ± 0.10 | 8.70 ± 0.56           | 14.50 ± 0.66 | 52.87 ± 1.81 | 24.43 ± 0.85 |
| Mean             | 4.92                  | 1.33        | 0.48        | 3.93        | 8.32                  | 13.95        | 51.14        | 23.97        |

| Barley varieties | (c) Non-starch polysaccharide (%) |                 |
|------------------|-----------------------------------|-----------------|
|                  | Arabinoxylan                      | $\beta$ -glucan |
| Jau-87           | 8.21                              | 3.67            |
| Haider-93        | 8.35                              | 3.65            |
| Mean             | 8.28                              | 3.66            |

Table 2. Mean values of dietary fibre content of native and chemically modified varieties of barley (g/100 g)

| Treatments                | Jau-87       |              |              | Haider-93    |              |              |
|---------------------------|--------------|--------------|--------------|--------------|--------------|--------------|
|                           | SDF          | IDF          | TDF          | SDF          | IDF          | TDF          |
| Control                   | 4.73         | 12.40        | 17.13        | 5.70         | 12.00        | 17.70        |
| Extruded                  | 4.80 ± 0.03  | 11.32 ± 0.04 | 16.12 ± 0.02 | 5.79 ± 0.01  | 10.90 ± 0.02 | 16.69 ± 0.01 |
| Acid-treated              | 5.85 ± 0.01  | 10.95 ± 0.01 | 16.8 ± 0.04  | 7.09 ± 0.04  | 10.59 ± 0.03 | 17.68 ± 0.01 |
| Acid-treated extruded     | 7.33 ± 0.03  | 11.91 ± 0.03 | 19.24 ± 0.01 | 8.75 ± 0.01  | 11.51 ± 0.03 | 20.26 ± 0.01 |
| Alkaline-treated          | 11.50 ± 0.12 | 11.77 ± 0.01 | 23.27 ± 0.02 | 13.90 ± 0.06 | 11.35 ± 0.03 | 25.25 ± 0.02 |
| Alkaline-treated-extruded | 12.45 ± 0.02 | 11.21 ± 0.01 | 23.66 ± 0.01 | 15.08 ± 0.02 | 10.51 ± 0.01 | 25.87 ± 0.02 |
| Alkaline-acid-treated     | 9.68 ± 0.02  | 11.52 ± 0.01 | 21.2 ± 0.17  | 11.63 ± 0.02 | 11.17 ± 0.02 | 22.77 ± 0.12 |
| Acid-Alkaline treated     | 41.22 ± 0.03 | 5.78 ± 0.03  | 47 ± 0.04    | 49.61 ± 0.03 | 5.64 ± 0.05  | 55.25 ± 0.04 |

TDF= total dietary fibre; IDF= insoluble dietary fibre; SDF= soluble dietary fibre

### Dietary fibre content of barley varieties

The soluble and insoluble fibre contents of the unmodified and all the chemically modified barley of two different varieties were measured.

Mean values for dietary fibre content of two barley varieties before treatments are exhibited in Table 2. Results showed that the soluble dietary fibre content was higher in Haider-93 (5.70 g/100 g dm) than in Jau-87 (4.73 g/100 g dm) whereas insoluble dietary fibre was more in Jau-87 (12.40 g/100 g dm) than in Haider-93 (12.40 g/100 g dm). Literature showed similar results to the ones presented in the present work. Beloshapka *et al.* (2016) explicated that barley contained about 8.6%, 4.8% and 13.4% insoluble, soluble and total dietary fibre, respectively.

Table 2 shows the mean values of dietary fibre content in chemically modified varieties of barley. Results of extrusion cooking showed that it slightly modified the soluble and insoluble dietary fibre ratio *i.e.*, soluble dietary fibre slightly increased while insoluble dietary fibre slightly decreased in both barley varieties. In Jau-87, soluble dietary fibre increased from 4.73 to 4.80 g/100 g, and insoluble dietary fibre decreased from 12.40 to 11.32 g/100 g, whereas in Haider-93, the increase in soluble dietary fibre was from 5.70 to 5.79 g/100 g, and the decrease in insoluble dietary fibre was from 12.00 to 10.90 g/100 g. However, this modification was not significant ( $P > 0.05$ ). Moreover, when the dietary fibre was treated with acid, it significantly modified the soluble and insoluble dietary fibre ratio *i.e.*, 23.68% increase in soluble and 11.69% decrease in insoluble dietary fibre in Jau-87, while in Haider-93, 24.39% increase in soluble and 11.75% decrease in insoluble dietary fibre, respectively. After treatment with acid, the dietary fibre was then extruded and it yielded much better results *i.e.*, in Jau-87, 54.97%

increase in soluble and 3.95% decrease in insoluble dietary fibre, while 53.51% increase in soluble and 4.08% decrease in insoluble dietary fibre in Haider-93 as shown in Figures 1(a), 1(b), 2(a), 2(b).

Furthermore, after acid and acid-extrusion treatments, dietary fibre was then treated with alkali (NaOH). This treatment significantly modified the soluble and insoluble ratio. In Jau-87, soluble dietary fibre significantly increased from 4.73 to 11.50 g/100 g and insoluble dietary fibre significantly decreased from 12.40 to 11.77 g/100 g, while in Haider-93, soluble dietary fibre significantly increased from 5.70 to 13.90 g/100 g and insoluble dietary fibre significantly decreased from 12.00 to 11.35 g/100 g. Then, this alkali treated dietary fibre was extruded, and the results were significant ( $P \leq 0.05$ ). Soluble dietary fibre increased from 4.73 to 12.45 and from 5.70 to 15.08 g/100 g while insoluble dietary fibre decreased from 12.40 to 11.21 and 12.00 to 10.51 g/100 g in Jau-87 and Haider-93, respectively.

After that, alkaline and acid treatment was applied simultaneously in two sequences (*i.e.*, alkaline-acid treatment, acid-alkaline treatment). The results of acid and alkaline treatments were significant ( $P \leq 0.05$ ). Alkaline-acid treatment increased 104.65 and 104.04% soluble dietary fibre and decreased 7.10 and 6.92% insoluble dietary fibre in Jau-87 and Haider-93, respectively, whereas acid-alkaline treatment increased the soluble dietary fibre to 771.46% whereas the decrease in insoluble dietary fibre was 53.39% (Figure 1, 2, 4, 5).

Among all treatments, the highest results were shown by acid-alkaline treatment. Extrusion cooking applied high temperature pressure and shear force and gasified and extended the moisture content present in both varieties of barley (Zhou *et al.*, 2012). It led to the modification of inter-molecular

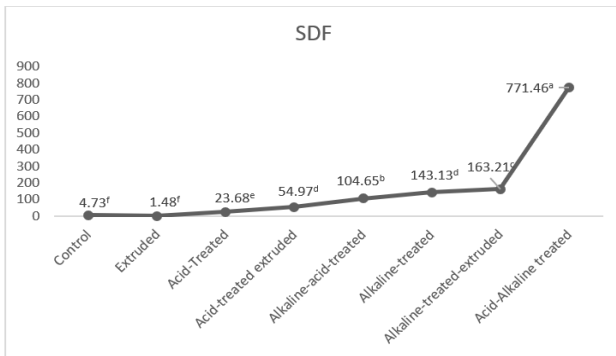


Figure 1 (a): SDF contents of chemically modified Jau-87

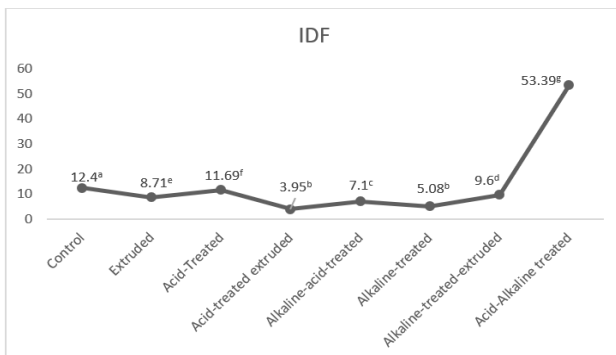


Figure 1 (b): IDF contents of chemically modified Jau-87

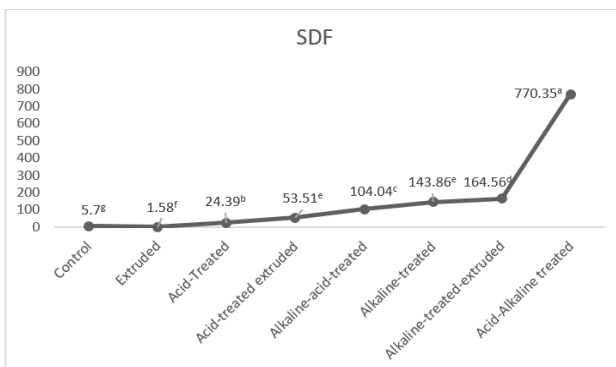


Figure 2 (a): SDF contents of chemically modified Haider-93

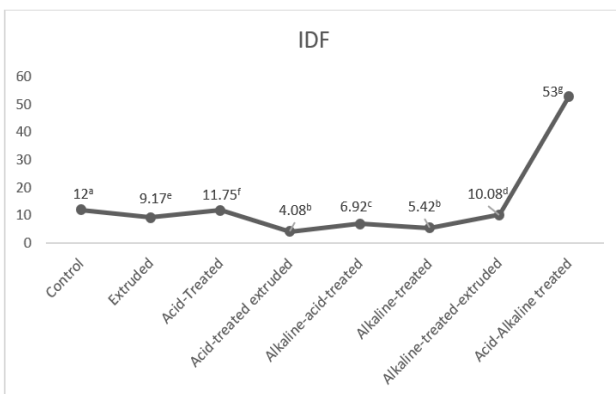


Figure 2 (b): IDF contents of chemically modified Haider-93

and intra-molecular spatial structure of fibre in barley and formed a porous state. Extrusion resulted in disruption of covalent and non-covalent bonds in the carbohydrate and protein moieties leading to smaller and more soluble molecular fragments and less insoluble fractions. Other chemical treatments (acid-treated, acid-treated extruded, alkaline-treated, alkaline-treated-extruded, alkaline-acid-treated, acid-alkaline treated varieties of barley) followed the same mechanism of polysaccharides chains breakage for the modification of soluble and insoluble dietary fibre. Hence, the mobility of the polysaccharide molecules was greatly increased, facilitating their interaction with water molecules and their associated solubility. The reason behind the extreme effectiveness of acid-alkaline treatment might be due to the fact that acid treatment can open the structure and increase the surface porosity of the fibre particle, making it easier for the hydroxyl groups to penetrate inside and perform hydrolysis during the subsequent alkaline treatment.

## Conclusion

Both varieties of barley were found to be very high-quality reservoir of protein and potassium and tremendous resource of dietary fibre mainly insoluble dietary fibre. Through chemical and extrusion application, the barley was modified with respect to soluble dietary fibre and insoluble dietary fibre ratio *i.e.*, soluble dietary fibre was increased and insoluble dietary fibre was decreased. Although all treatments had given effective results, acid-alkaline treatment was found the most effective. This modification opens the door for the betterment of physiochemical, physiological, and functional properties of dietary fibre by increasing the soluble dietary fibre. As a promising source of soluble dietary fibre, barley should be exploited for therapeutic and health enhancing food products.

## Acknowledgement

The Higher Education Commission, Government College University, Institute of Home and Food Sciences were acknowledged for providing funds, facilities and equipment.

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